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A NOTE ON HYDROCYANIC ACID IN THE BURMA BEAN (*PHASEOLUS LUNATUS* SP.)

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PREFACE

Owing to my transfer from Burma to my present post I was unable to carry out the programme of work I had undertaken. The results obtained up to the date of my departure are recorded in this note in the hope that they may be of assistance in the further prosecution of the work.

F. J. Warth.

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[Received for publication on the 12th July, 1922.]

I. The determination of HCN.

THAT the determination of HCN in plant products is not a straightforward matter may be gathered from the voluminous literature on this subject. Two examples will suffice to show the nature of the difficulties which may be met with. Alsberg and Black (*Jour. Biol. Chem.*, 1916) found that leaves of *Prunus Virginiana* must be distilled with acid for four hours in order to liberate all the HCN, whereas one hour is sufficient for *Andropogon*. They found also that by macerating plant tissues containing HCN a certain amount of the HCN present or of cyanide added is so converted that it cannot be recovered by distillation with H_2SO_4. They considered this was not due either to enzymes or to the presence of glucose. They recommended that in determining HCN in plants several methods should be employed to corroborate one another. Viehover Johns and Alsberg (*Intern. Bull.*, 1918) working with *Tridens flavus* found that maceration followed by distillation with acid gave lower results than immediate distillation with acid. Distillation of the macerated or unmacerated plant without acid resulted in a partial or complete loss of the available HCN.

In my experiments with the Burma bean macerated and unmacerated leaf gave identical results. Acid also gave no increased yield in conjunction with the enzyme process. The procedure to be adopted must, therefore, be varied according to the plant under investigation.

The determination of HCN in the Burma bean offers its own peculiar difficulties however; but before entering into these, the analytical methods employed will be briefly outlined.

1. *Auto-enzyme hydrolysis.* The material—macerated or ground if this preliminary treatment appeared to be necessary—was soaked in water for 24 hours generally. The HCN liberated by this treatment was distilled off and absorbed in alkali. The distillation was usually effected by means of steam and the HCN determined as a rule by precipitation as Prussian blue.

2. *Extraction and separation of the crude glucoside with alcohol and its subsequent hydrolysis with* H_2SO_4. This process was carried out on the lines laid down by Dunstan and Henry.

3. *Extraction with hot water.* For Burma beans I have found that extraction with boiling water is as effective as and more convenient than the alcohol extraction. The third method therefore consisted in extracting the glucoside with boiling water and hydrolysing the extract with acid. In this process it must be noted that when dealing with material containing active enzyme, such as the leaf, some liberation of HCN is to be expected at the moment of plunging the leaf into boiling water. This was found to be the case very frequently.

The HCN so formed (it will be spoken of as free HCN for the sake of brevity) had to be distilled off and determined separately before proceeding with the extraction and hydrolysis. In the water extraction process generally 2 figures will be given, namely :—

(*a*) Free HCN.

(*b*) HCN by acid hydrolysis.

It should be understood that these two figures combined give the HCN obtainable by this method. The free HCN is, however, generally negligible in amount.

As the acid hydrolysis was required for both the second and third processes it has been thoroughly tested to arrive at the best conditions of working.

The following procedure was finally adopted as yielding the highest results in the time available for completing the determination in one day.

The extract made up to 300 c.c., containing 50 c.c. conc. H_2SO_4, was placed in a flask and connected to a condenser at the delivery-end of which an alkali

scrubber was attached. Hydrolysis was allowed to proceed by heating the flask in a vigorously boiling water bath. At the end of two hours and again at the end of four hours a rapid current of steam was passed through the hydrolysing liquor to remove all free HCN to the absorbing bottle. The hydrolysis was stopped at the end of six hours and a final passage of steam employed to remove the last traces of HCN.

The graph (Fig. 1) shows that by this procedure practically all the determinable HCN can be liberated in six hours.

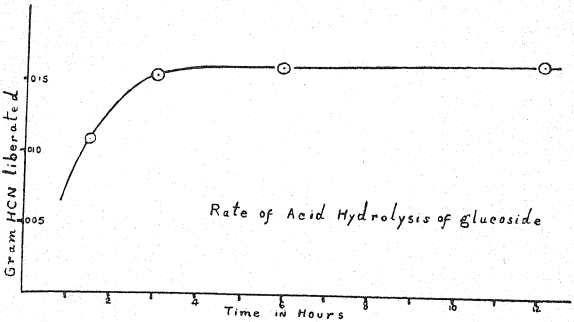

FIG. 1.

4. *Enzyme powder.* An extract having been prepared according to method (3) or other convenient procedure it was treated with enzyme powder and allowed to stand for 24 hours in a closed flask. The HCN liberated by the action of the added enzyme was then distilled off and determined as in the first method.

For the determination of HCN in the seed and plant parts of the Burma bean in almost every instance at least two of the above methods were employed.

It may be said at once that concordant results were never obtained; but a certain regularity in the discrepancies revealed itself at an early stage of the work, and this regularity in the discrepancies eventually explained some of the difficulties, and called attention to the need for a study of enzyme activity.

It was found for example that when dealing with fresh leaf the figure obtained by the auto-enzyme process was invariably higher than that obtained by the complete acid-hydrolysis method (including free HCN).

On the other hand determination of HCN in the green pod gave equally regularly the opposite result (namely, a low auto-enzyme figure and a high acid-hydrolysis figure).

It is unnecessary to quote data here. Typical examples will be found in most of the subsequent tables of analysis.

The peculiar results described above are now known to be due to differences in enzyme activity in these two types of tissue. The hydrolysing enzyme in the pod is very weak and therefore the auto-enzyme process when applied to the pod yields very little HCN. The green leaf which contains an active hydrolytic enzyme yields high results with the auto-enzyme process. In this case, however, there is no doubt that the acid-hydrolysis process is very inefficient and therefore we cannot assume that it is more efficient when applied to the pod although with the pod it yields higher results than are obtained by the enzyme.

The acid-hydrolysis process therefore as a means for determining HCN cannot be considered satisfactory. But when used in conjunction with the auto-enzyme process it yields valuable information regarding enzyme activity and has been used with success for this purpose throughout the present enquiry.

Losses of HCN in the acid-hydrolysis process. As the process was of value a number of experiments were carried out to determine where and how the losses involved in it occur. These experiments will be discussed next :—

(1) A crude glucoside extract prepared from leaf was divided into two parts, one part was submitted to acid-hydrolysis (method 3) and the other treated with enzyme powder (method 4). The results obtained were as follows :—

	Grm. HCN
HCN by acid-hydrolysis ..	.. 0·0038
,, enzyme powder ..	.. 0·0033

The test was made on several occasions but did not yield uniform results ; one test, for example, gave the following result :—

	Grm. HCN
HCN by acid-hydrolysis ..	.. 0·0056
,, enzyme powder ..	.. 0·0074

The efficiency of the enzyme powders could not be doubted. The lack of uniformity in the results could only be attributed to differences in behaviour of the different glucoside preparations.

(2) Losses which might occur during concentration of the aqueous extract preparatory to acid-hydrolysis were looked for.

An extract of crude glucoside was divided into four parts. Two of these were hydrolysed at once. The other two were diluted to double their volume, again concentrated down to their former volume and hydrolysed.

The four determinations yielded absolutely identical results.

Concentration therefore did not cause loss of any substance capable of yielding HCN by acid-hydrolysis.

Another experiment was carried out as follows: An extract was divided into two parts. One part was treated at once with enzyme powder. The other part was concentrated and then submitted to acid-hydrolysis. The following results were obtained :—

	Grm. HCN
HCN by acid-hydrolysis	.. 0·0076
,, enzyme powder ..	.. 0·0120

This experiment indicates that the liquor contains a substance which yields HCN to enzyme powder but not to acid-hydrolysis.

(3) To determine whether any HCN is distilled off during concentration, the entire extraction and concentration was carried out in flasks connected to condensers. The distillates were collected fractionally and examined for HCN. No trace of HCN was found in the distillates.

Loss of HCN was nevertheless proved to have taken place in this experiment. The original leaf contained 0·0270 per cent. HCN. Extraction and treatment of the extract with enzyme powder (to avoid all suspicion of loss through inefficiency of acid-hydrolysis) yielded 0·0195 per cent. HCN. Some free HCN was evolved at the commencement of the experiment. This amounted to 0·001 per cent. HCN. Out of a total of 0·027 per cent., therefore, 0·006 per cent. remains unaccounted for.

The loss in this case is not serious. It may amount to half the HCN of the leaf when acid-hydrolysis instead of enzyme powder is used. The experiments had to stop at this stage.

As far as they go they indicate that part of the loss at least is due to the presence in the extract of a substance which cannot yield HCN by acid-hydrolysis, but is able to do so when treated with enzyme powder.

There remains an observation in connection with other work which may be suitably quoted here.

It refers to fresh leaf in which the acid-hydrolysis process compares most unfavourably with the auto-enzyme method.

On three separate occasions after the HCN had been liberated as completely as possible by the auto-enzyme process and distilled off by steam, the residual liquor was concentrated by boiling and treated by the acid-hydrolysis process. A small but determinable amount of HCN was obtained in each case (by precipitation as Prussian blue). That is to say, the small residues of glucoside remaining after a vigorous enzyme has ceased to act, can be easily detected and determined by the water extraction and acid-hydrolysis process. The delicacy of the process therefore cannot be doubted.

We have to conclude that when fresh leaf is plunged into boiling water a certain amount of glucoside is converted into a substance which cannot yield HCN by acid-hydrolysis.

The effect, though not identical with, bears a resemblance to the observations of Viehover Johns and Alsberg with other plants.

Finally some remarks regarding the fourth process are called for.

When the total HCN content of an organ has to be determined the process has undoubted advantages over the acid-hydrolysis method.

In the first place concentration of the extract is not necessary—thus avoiding possible loss in this way. It is not even necessary to separate the plant part from the extract. The enzyme powder may be added as soon as the enzymes present in the organ under examination have been killed. The last precaution is undoubtedly necessary at times. An example illustrating this point is given later.

That it is able to liberate HCN in some form which cannot be liberated by the acid process has already been noticed.

II. Enzyme transformation of the glucoside and of HCN during drying of the leaf.

A. Hydrolytic power of fresh and sun-dry leaf.

The evolution of the so-called free HCN which has already been referred to under the water extraction method became prominent during the preparation and study of enzyme powder for the fourth method of estimating HCN.

The dried powder before extraction with alcohol was submitted to the systematic tests made on all plant tissues (*viz.*, determination of free HCN ; acid-hydrol. HCN ; and auto-enzyme HCN). This dry leaf was found to contain :—

(*a*) less total HCN than the original.

(*b*) a much higher proportion of free HCN than the original.

The following figures are typical of such sun-dried material :—

TABLE I.

Free HCN from fresh and sun-dried leaf.

	Free HCN	HCN by acid-hydrol.	HCN by enzyme
Fresh leaf 36 grm. ..	0·0020	0·0088	0·0200
Corresponding sun-dried leaf. ..	0·0086	0·0066	0·0150

Ravenna (*Jour. Chem. Soc.*, 1912, A. 2, 798, etc.) who has carried out a large number of tests on the evolution of free HCN by leaves when plunged into boiling water considers the phenomenon to be due to enzyme action during the short period of heating up required to kill the enzyme. No doubt this is generally the case, but if it were so in the present instance our sun-dried leaf should yield an extraordinarily active enzyme, seeing that its production of free HCN is strikingly high when compared with the fresh leaf.

As the primary question at that time was to procure an active enzyme powder, experiments were undertaken to compare the hydrolytic powers of enzyme powder prepared from fresh and sun-dried leaf respectively.

The experiment was carried out by extracting the tissue with alcohol and immediately using the material thus obtained for hydrolysing a crude glucoside solution. To make allowance for any deleterious effect of the alcohol upon the enzyme a series of enzyme powders, receiving increasing alcoholic treatment, was prepared from identical tissue, and the activity of each sample determined. The following results were obtained :—

TABLE II.

Effect of sun-drying on the activity of the hydrolytic enzyme.

Alcohol treatment	Time of action	GRM. HCN LIBERATED BY	
		Fresh leaf enzyme *	Sun-dried leaf enzyme *
	Mts.		
12 hrs.	30	0·0040	0·0012
	60	0·0072	0·0054
24 hrs.	30	0·0052	0·0040
	60	0·0130	0·0030
36 hrs.	30	0·0058	0·0050
	60	0·0102	0·0076
48 hrs.	30	0·0026	0·0020
	60	0·0098	0·0062

* In all cases quantity of enzyme is equivalent to 12 grm. fresh leaf.

These tests show in the first place that sun-dry enzyme is not more active than fresh leaf enzyme.

The figures also prove in a remarkable manner that alcohol is not injurious up to a certain point. It appears in fact that by continued soaking in alcohol the activity of the enzyme increases, reaches a maximum and then diminishes.

As it was intended to use this enzyme freely a further experiment to prove this point was undertaken. The experiment was carried out exactly as before, in this case young and old leaves being compared. Results obtained were as follows :—

TABLE III.

Comparison of hydrolytic power of young and old leaves.

Alcohol treatment	Time of reaction	HCN LIBERATED BY ENZYME OF			
		YOUNG LEAF		OLD LEAF	
		Fresh	Sun-dry	Fresh	Sun-dry
	Mts.				
12 hrs.	30	0·0030	0·0034	0·0030	0·0010
	60	0·0066	0·0068	0·0076	0·0038
24 hrs.	30	0·0106	0·0044	0·0050	0·0018
	60	0·0132	0·0076	0·0092	0·0050
36 hrs.	30	0·0054	0·0038	0·0030	0·0030
	60	0·0102	0·0074	0·0070	0·0068

Leaf enzyme powder used in each test was equivalent to 12 grm. fresh leaf. There is little doubt that the above-noted effect of alcohol is real. The point may be of interest to workers on plant enzymes. The experiments have established that young leaf is somewhat more active than old leaf; and fresh leaf more active than sun-dry leaf.

The greatly increased evolution of free HCN by sun-dried leaf is therefore not due to increased enzyme activity but to a partial decomposition of the glucoside during sun-drying. The form in which the decomposition product exists in the leaf before boiling out is not known. Further experiments on this free HCN will be described below.

B. Losses of HCN during sun-drying of leaves.

Loss of HCN from leaves of cyanogenetic plants during drying has frequently been remarked upon.

Ravenna and Tonegutti (*J. C. S.* 1910, A. 2, 884) found that the loss was much greater when leaves were dried slowly at the ordinary temperature than when dried at 130°F. They remarked also that no HCN is evolved during the process.

Couperot (*J. S. C. I.*, 1909, p. 219) working with *Sambucus* found that drying led to simultaneous loss of nitrate and HCN. If the plant was dried quickly by heating to 60°F. there appeared to be no loss.

That HCN is lost during the drying of the leaves of Burma bean has been noted above.

A series of jar experiments was now undertaken to determine whether HCN as such is emitted during the process.

The apparatus consisted of large specimen jars into which wire trays were fixed. The air inlet consisted of a glass tube passing through the lid to the bottom of the jar. The exit tube drew air from the top of the jar and was connected through two glass bead alkali scrubbers, in series, to the water pump.

The efficiency of the apparatus for carrying off and absorbing HCN was tested as follows :—

Two jars with separate connections and absorbing tubes having been fitted up, the air current was turned on. Into the first jar on each of four consecutive days 1 c.c. of standard KCN solution was dropped on to a sheet of acidified filter paper. Into the second jar 4 c.c. were added at once. The aspiration was carried out continuously for five days. At the end of this time the HCN in the scrubbers and jars was determined.

The following figures were obtained :—

TABLE IV.

Efficiency test of jar aspiration and absorption of HCN in scrubbers.

	1st experiment	2nd experiment
	Grm. HCN	Grm. HCN
1st scrubber	0·0056	0·0038
2nd scrubber	0·0000	Trace.
Residue in jar..	0·0006	0·0018*

* Some water got into this jar and prevented evaporation of HCN.

Therefore for a steady evolution of HCN the apparatus is perfectly satisfactory.

Leaf was next dried in these jars, and no trace of HCN was obtained in the scrubbers. The test showed that under the conditions of this experiment HCN is not evolved by drying leaves.

The dry leaf produced in this way was next examined.

It was found to yield a small amount of free HCN and none whatever by auto-enzyme action.

The product obtained by this drying process is therefore very peculiar and differs entirely in its properties from the sun-dried material. The lack of auto-enzyme effect may be contrasted with the figures for sun-dried leaf in Table I. As the material produced was so unlike that which was sought, the laboratory jar experiment was abandoned for the time being, and a new apparatus set up for drying leaf under conditions similar to those which produced the original sun-dry powder.

To accomplish this the leaves were placed on a cement floor exposed to full sunlight, and covered by large bell jars. The jars were sealed to the floor by means of a thick layer of bees-wax through which two tubes were passed as inlet and outlet pipes respectively.

With the rapid drying of the leaves under these conditions a powerful air current was necessary to prevent condensation of moisture on the jars. Four sets of scrubbers had to be employed in parallel to give a sufficient passage for the large volume of air aspirated during the experiment.

When the leaves looked dry the experiment was stopped and the absorbing liquid in the scrubbers examined. It was found to contain an appreciable amount of HCN.

In this experiment therefore the leaves during drying had undoubtedly evolved HCN.

The experiment was repeated using young leaves (vigorous enzyme) and older leaves (less vigorous enzyme) and gave the following results :—

TABLE V.

Evolution of HCN by sun-drying.

	Young leaf	Older leaf
	Grm.	Grm.
Weight of fresh leaf	30·0	30·0
Weight after 5 hrs. sun-drying under bell jars	6·9	6·2
HCN evolved and obtained in scrubbers	0·0065	0·0020
HCN content of dried leaf — Free HCN (by boiling)	0·0017	0·0015
HCN content of dried leaf — HCN by acid-hydrol.	0·0007	0·0020

The figures indicate that fresh leaves whether young or old invariably evolve HCN when sun-dried, and the quantity so evolved may amount to half the HCN content of the leaf. The rate of evolution evidently depends upon the initial activity of the enzyme—the young leaf evolving decidedly more than the older leaf.

It will be observed too that the glucoside (*i.e.*, HCN by acid-hydrolysis) in the young leaf has been almost completely exhausted during the process, whilst in the old leaf an appreciable amount remains unconsumed.

One experiment was made to determine the time taken for the initiation of this change in the leaf.

Two lots of leaf were placed under jars as usual and aspirated. The scrubbers were renewed at two-hour intervals and the HCN evolved during these two-hour intervals determined.

The following figures were obtained in the two tests :—

TABLE VI.

Rate of HCN evolution during sun-drying.

	A	B
	Grm.	Grm.
Weight of leaf used	15.0	15.0
HCN evolved during .. 1st 2 hrs.	0·0003	0·0000
HCN evolved during .. 2nd 2 „	0·0022	0·0017
HCN evolved during .. 3rd 2 „	0·0012	0·0019
TOTAL ..	0·0037	0·0036
FINAL WEIGHT OF LEAF ..	3·2	3·2

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The figures indicate that fresh leaves whether young or old invariably evolve HCN when sun-dried, and the quantity so evolved may amount to half the HCN content of the leaf. The rate of evolution evidently depends upon the initial activity of the enzyme—the young leaf evolving decidedly more than the older leaf.

It will be observed too that the glucoside (*i.e.*, HCN by acid-hydrolysis) in the young leaf has been almost completely exhausted during the process, whilst in the old leaf an appreciable amount remains unconsumed.

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		Grm.	Grm.
Weight of leaf used		15.0	15.0
HCN evolved during	1st 2 hrs.	0·0003	0·0000
	2nd 2 „	0·0022	0·0017
	3rd 2 „	0·0012	0·0019
	TOTAL	0·0037	0·0036
	FINAL WEIGHT OF LEAF	3·2	3·2

The evolution takes about two hours to commence. In the one case it was evidently coming to an end in six hours. In the other case it is not possible to say.

Owing to the time occupied by the necessary preliminary preparations for the experiment more than six hours' sun-drying could not be given. Further changes were therefore looked for by treating sun-dry leaf to aspiration in jars in the laboratory as in the preliminary trial.

For this purpose 150 grm. of fresh leaf were sun-dried for six hours. Weight after completion of this drying was 45 grm. The sample was divided into two parts, one part being placed at once in an aspiration jar, the other part being used for analysis. Laboratory aspiration with dry air in this case yielded a large amount of HCN. Finally the aspirated leaf was analysed. The following results were obtained :—

TABLE VII.

Jar aspiration of sun-dry leaf.

		Jar aspiration	Immediate analysis
		Grm.	Grm.
Weight of sun-dry leaf used		22·5	22·5
Weight after 4 days' aspiration		15·4	..
HCN in scrubbers ..	1st period of 2 days	0·0065	..
	2nd period of 2 days	0·0000	..
HCN in leaf ..	Free	0·0029*	0·0053†
	Acid-hydrol.	0·0032*	0·0071†
	TOTAL HCN	0·0126	0·0124

* After the aspiration treatment.
† Initial.

In this case the HCN has been completely accounted for. It will be found on reference to other data that this is invariably the case with sun-dry leaf. This drying process apparently liberates all the HCN existing in a form liable to destruction by boiling water.

It may also be noticed here that some glucoside was hydrolysed during the aspiration (acid-hydrol. figure has been very materially reduced). The leaf apparently contained sufficient moisture for this enzyme reaction.

It is most probable therefore that much of the HCN evolved during this aspiration period was also liberated by enzyme action set in motion during sun-drying. Sun-drying therefore directs the vital processes towards liberation of free HCN. In addition to this, however, the high "free HCN" content of the sun-dry leaf shows that it contains bodies which decompose in boiling water with evolution of HCN in a way which the glucoside of the fresh leaf does not.

C. Slow air-drying of leaf.

HCN having been obtained so readily in the last experiment by the laboratory jar aspiration method, this process was once more applied to fresh leaf which, it will be remembered, had given a negative result in the preliminary test.

This time the experiment was carried out simultaneously with a blank empty jar to which HCN was added. At the end of four days a complete recovery of HCN from the blank was obtained and again no trace from the leaf. It is certain therefore that leaf dried under these conditions does not evolve HCN.

The leaf after this treatment was thoroughly examined. It yielded a small amount of free HCN but zero by the auto-enzyme process. The leaf therefore had lost practically all its HCN, though none was evolved.

The fact that the leaf yields a little free HCN but none by the auto-enzyme process is remarkable. It shows that even the small residue of free HCN it contains disappears during the auto-enzyme process.

Assuming that the leaf had lost its hydrolytic power it was next treated with a very active enzyme powder for determination of HCN by the fourth process described above.

The enzyme powders used in this work were all prepared by extracting fresh leaf with alcohol. They are never quite free of HCN and a blank determination is always made to allow for HCN in the enzyme powder.

The test with this jar-dried leaf was carried out in duplicate, two flasks containing leaf and enzyme powder, two flasks enzyme powder alone. The flasks with enzyme powder alone yielded the usual amount of Prussian blue, but the flasks with leaf and enzyme powder gave no trace of colour.

This unexpected result was the first indication that HCN is destroyed in the presence of slowly dried leaf.

The experiment may also be noted as a case in which the enzyme powder process fails unless the enzymes existing in the tissue to be examined are destroyed before addition of the enzyme powder.

The total HCN content of this leaf as determined by boiling and extraction was—

Free HCN .. .. trace.
HCN by acid-hydrol. .. .. .. trace.

The original HCN in the leaf had therefore disappeared almost completely and none whatever had been evolved as gas.

Under conditions of slow air-drying therefore the HCN of the leaf appears to undergo enzymic auto-digestion.

A quantity of leaf was next slowly air-dried in the laboratory to determine whether such leaf possesses the power of destroying HCN. The following experiment was carried out with the material prepared in bulk :—

Into each of 8 flasks 5 c.c. KCN solution were measured. The liquids were made up to 50 c.c. in four of these and to 200 c.c. in the others. Six of the flasks were each treated with 4 grm. of leaf powder. Two flasks were treated with leaf powder previously boiled in water. The contents were then mixed, the flasks closed and allowed to stand overnight. Next morning the HCN was distilled off and determined as usual. The following results were obtained :—

TABLE VIII.

Destruction of HCN by slowly dried leaf.

	Contents of flask	Grm. HCN obtained
1.	Four grm. leaf and 45 c.c. water and 5 c.c. KCN	0·0020
2.	Ditto	0·0020
3.	Ditto leaf boiled	0·0064
4.	Four grm. leaf and 195 c.c. water and 5 c.c. KCN	0·0020
5.	Ditto	0·0020
6.	Ditto leaf boiled	0·0070
7.	Five c.c. KCN det. in concentrated solution without distillation	0·0090
8.	Ditto	0·0088

In all cases the leaf powder led to loss of HCN. The boiled leaf caused some loss, part of which is attributable to unavoidable loss of a trace in distillation.

It should be noted too that boiling of the leaf drives off the small amount of free HCN it contains. The flasks of boiled leaf therefore began with 0·002 grm. less HCN than the other flasks.

Destruction of HCN by unboiled leaf is unmistakably shown by these figures. The leaf itself was submitted to the systematic tests and yielded the following figures :—

	Grm.
Free HCN	0·0022
Acid-hydrol.	0·0008
Auto-enzyme	0·0000

The leaf shows again the typical effect of destroying the small amount of free HCN it contains when the auto-enzyme process is attempted with it.

The above experiment was next carried out in a modified form to determine the effect on glucoside as well as on HCN. Three flasks containing HCN and 3 flasks containing glucoside were used for the test. The experiment was carried out exactly as above with this difference that after distilling off the HCN as usual the residual liquor was submitted to acid-hydrolysis to determine any residual glucosidic HCN. The following results were obtained :—

TABLE IX.

Destruction of free and glucosidic HCN by slowly dried leaf.

	Grm. HCN by dist.	Grm. residual glucosidic HCN
1. Leaf powder + 5 c.c. KCN	0·0028	0·0008
2. Ditto	0·0024	0·0008
3. Boiled leaf + 5 c.c. KCN	0·0064	0·0010
4. Leaf powder + glucoside	0·0022	0·0008
5. Ditto	0·0022	0·0008
6. Boiled leaf + glucoside	0·0004	0·0054

The experiment corroborates the previous results and shows that the properties of the leaf, which cause it to lose the HCN it contains, persist in the dry leaf and are able to cause destruction of HCN both free and glucosidic.

The boiled leaf powder naturally cannot in any way affect the glucoside. The fact that HCN was still present in this case was revealed by acid-hydrolysis.

The experiment is striking, and if the determinations are made by precipitating the HCN as Prussian blue, gives a convincing ocular demonstration.

It may be noted that this slowly-dried leaf of the Burma bean behaves in a manner similar to the fresh tissue of *Tridens flavus* and other plants examined by Viehover Johns and Alsberg (*loc. cit.*).

D. Effect of fresh leaves on HCN.

Up to this stage the fresh leaf has never shown any indication of a destructive effect on HCN, but in view of the last result and the experience of workers with other plants it seemed desirable to carry out a more careful examination of this point.

The test was made as follows :—

Three solutions containing respectively 0·2286, 0·1740, 0·0870 grm. HCN per 1000 c.c. were prepared. Lots of 200 c.c. from each of these solutions were placed in bottles, and one lot of bottles with 200 c.c. water only was prepared. The bottles were brought to constant temperature in a bath maintained at 30°C. 30 grm. of fresh leaf were introduced into each bottle and the bottles replaced in the bath. The contents of the bottles were shaken at regular intervals. At the expiry of 2, 4 and 8 hours one bottle of each series was taken out, the liquid decanted, made alkaline to stop enzyme action and an aliquot heated, acidified and distilled. The HCN in the distillate was determined by iodine titration. The figure thus obtained includes the HCN originally added and that obtained from the leaf. By subtracting the initial HCN content of the liquor the HCN derived from the leaf is obtained.

In this way the HCN liberated from the leaf during each period was determined. The results obtained were as follows :—

Table X.

Effect of HCN solutions on the evolution of HCN by fresh leaf.

		A	B	C	D
Initial HCN content of soln. in grm. ..		0·04572	0·03480	0·0174	0·0000
HCN in grm. evolved by leaf during	1st period 2 hours	0·00003	0·00117	0·01124	0·00900
	2nd period 2 hours	0·01545	0·01770	0·00742	0·01110
	3rd period 4 hours	0·00780	0·01020	0·01020	0·01770
Total HCN evolved by leaf in grm. ..		0·02328	0·02907	0·02886	0·03780

The initial HCN content of the liquor is seen from these figures to have a marked effect upon the rate of evolution of HCN by the leaf. The total HCN evolved during the period is similarly influenced. The figures do not tell us

however whether the reaction had come to an end. To settle this point another set of determinations had to be undertaken. It yielded the following results :—

TABLE XI.

Effect of HCN solutions on the evolution of HCN by fresh leaf.

Initial HCN content of solution in grm.		A. 0·03396		B. 0·00000	
		Total HCN	HCN from leaf	Total HCN	HCN from leaf
Grm. HCN after	2 hrs.	0·04116	0·00720	0·00742	0·00742
In sol.	„ 4 hrs.	0·04792	0·01396	0·01935	0·01935
	„ 8 hrs.	0·05355	0·01959	0·02520	0·02520
	„ 16 hrs.	0·05200	0·01804	0·02430	0·02430
	„ 24 hrs.	0·05290	0·01894	0·02430	0·02430

The leaf is not identical with that used above but evidently under such conditions HCN evolution ceases entirely within a period of 8 hours.

Knowing that the reaction ceases in 8 hours it is possible from the results of Table X to draw curves showing roughly the velocity of HCN evolution from the leaf. These are shown below in Fig. 2.

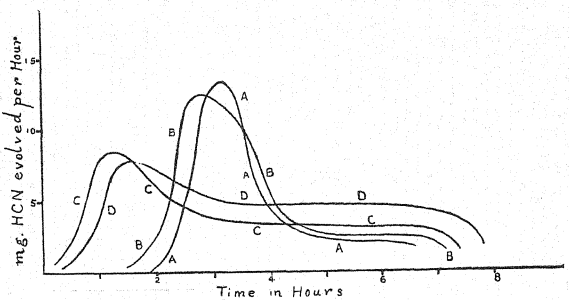


FIG 2. Showing velocity of HCN evolution.

Reference to the velocity graph brings out a number of points. In the first place it will be seen that when leaf is placed in relatively strong HCN solutions the evolution of HCN is very much retarded. It eventually comes out at a very great speed for a short period after which the velocity of evolution slowly dies down.

Another remarkable point brought out by this graph is that a weaker solution of HCN will accelerate the evolution—the initial speed in water being taken as our standard. The solution suitable to attain this result will no doubt depend upon the enzyme activity of the leaf. This is indicated by the fact that the results in the two tables from slightly different material, though similar, do not quite agree. From a combined examination of the graphs and the figures the following conclusions regarding this peculiar phenomenon may be arrived at :—

1. HCN in solution affects the permeability of the tissue. The permeability may be either increased or decreased according as the solution is weak or strong. The HCN in this case must play a part in regulating the vital processes in the leaf.
2. In the presence of HCN the hydrolysis ends prematurely. The total HCN obtained from the leaf being less in the presence of HCN than when water alone is used. This is apparently due to paralysis of the enzyme. This property of HCN must provide the plant with a delicate means for controlling hydrolysis.

The experiment does not reveal any definite sign of assimilation of HCN though it shows very clearly that the hydrolysis ends prematurely.

III. Enzyme activity of other plant parts.

In the young stalk the enzyme is sufficiently active to liberate large amounts of HCN by the auto-enzyme process. The usual test for enzyme activity, *viz.*, HCN determination by auto-enzyme action and by acid-hydrolysis gave in two samples the following results :—

TABLE XII.

Enzyme activity of young stalks.

	HCN FROM 36 GRM. FRESH STALK	
	By enzyme	By acid-hydrolysis
Sample 1	0·0054	0·0030
Sample 2	0·0078	0·0048

The auto-enzyme figure is decidedly higher than the figure by acid-hydrolysis which is taken as a proof of enzyme activity. This is not the case with all plant parts. The following table of analysis carried out on an early harvested crop shows the behaviour of various organs in this respect :—

TABLE XIII.

Analyses of crops harvested on 27th January, 1919.

Determination	HCN IN GRM. OBTAINED FROM 36 GRM. FRESHLY HARVESTED MATERIAL											
	Green leaf		Dead leaf		Stalk		Dry pod		Ripe seed		Green pod	
	Auto-enzyme	Acid-hydrol.	Auto-enzyme	Acid-hydrol.	Auto-enzyme	Acid-hydrol.	Auto-enzyme	Acid-hydrol.	Auto-enzyme	Acid-hydrol.	Auto-enzyme	Acid-hydrol.
Sample A, good seed ...	0·0136	0·0094	0·0010	0·0024	0·0076	0 0058	0·0010	0 0122	0·0000	0·0026	0·0016	0·0052
Sample B, bad seed ...	0·0076	0·0062	0·0006	0·0016	0·0036	0·0080	0·0004	0·0072	0·0016	0·0144	0·0002	0·0030

By good seed and bad seed is meant seed having low and high HCN content respectively.

The analyses were carried out immediately after the crop had been cut. The table contains a number of interesting points. It will be noticed for example that sample B is decidedly riper than sample A. The fresh leaves of B contain somewhat less HCN and the proportion of this recovered by acid-hydrolysis is higher than in A, which is a clear sign of decaying enzyme activity. These changes in the leaf have already been shown to accompany advancing maturity. The stalk too has advanced much further. It has lost much of its hydrolytic power—which is demonstrated again by the fact that the recovery of HCN by acid-hydrolysis is higher than by auto-enzyme action in sample B. The green pods are of considerable interest. They are the only part of the plant which in the perfectly green state is almost devoid of hydrolytic power—the amount of HCN liberated by auto-enzyme action being very small and forming also a very small proportion of the amount recoverable by acid-hydrolysis.

It was found that the pod begins to assume this character when the seeds commence to fill out. In December the young pods behaved like the leaves. There seems to be little change in HCN content of the pod during ripening. The relatively high figures for dry pod are mainly due to loss of moisture (as all figures in the table are based on 36 grm. of fresh sample).

By dead leaf is meant leaf which has become bleached and has just fallen.

In this case we are dealing with air-dry material comparable to the dry pods. The difference in HCN content of these two organs is striking.

The leaf during maturing (the figures for dead leaf should be divided by 4 to be compared with the fresh leaf) has lost almost all its HCN, whilst the pod has lost little or none. It is true this dead leaf gives higher HCN figures by acid-hydrolysis than by the auto-enzyme method, which proves that its hydrolytic power has been very much reduced. It is however still appreciable for the ratio of auto-enzyme HCN to acid hydrolysed HCN is not very low.

Judged by this criterion the hydrolytic power of the dry pod is negligible compared with the dead leaf.

Finally we come to the seed. The seed is also practically devoid of hydrolytic power and in this respect behaves similarly to the pod.

Concerning the fate of the HCN in the plant during ripening it may be concluded therefore that the leaf gradually parts with its HCN. Presumably this must pass in some form to the still vigorous parts of the plant.

A similar but less marked decrease takes place in the stem. The pod alone is peculiar in this respect, it does not discharge its HCN. It appeared possible from the results that the pod was assimilating N in the form of HCN. If so there was a possibility that it might show the phenomenon of auto-digestion.

Green and dry pods and dead leaf also were examined to test this point. The results of the test are given in—

TABLE XIV.

	Grm. HCN from 36 grm. fresh material		
	Old dead leaf	Late fresh husk	Green pod
Free HCN	0·0012	0·0003	0·0000
Acid-hydrol.	0·0050	0·0070	0·0050
TOTAL ..	0·0062	0·0073	0·0050
Enzyme HCN	0·0050	0·0006	0·0014
Followed by acid-hydrolysis of residue ..	0·0044	0·0066	0·0040
TOTAL ..	0·0094	0·0072	0·0054

There is no indication of assimilation in any of these cases. It should be explained that in this experiment no attempt was made to develop auto-

digestion power by slow drying or other means. The object was to determine whether the property was apparent in the organ in that state of development in which it was harvested.

We may note again that in the case of leaf—even of leaf which is virtually dead in this instance—there is an undetermined factor vitiating results. Here again the difference must be attributed to HCN compounds which are unstable in boiling water.

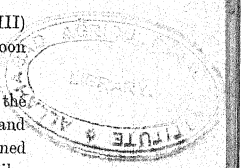
This process for discovering signs of assimilation was not applied to the seed ; but from what is now known of the seed it may be taken for granted that a negative result—similar to that observed in the case of the pod—would have been obtained.

A more refined test for auto-digestion has however since been applied to the seed. This will be described under seed experiments.

IV. Enzyme activity of seed in relation to age and storage conditions.

The study of enzyme activity in the plant has led to an important practical question. It supplied means for studying enzyme activity in the seed and also directed attention to this matter.

After the harvest experiments of January 1919 (given in Table XIII) had been completed the main facts relating to seed enzyme were very soon discovered.



In the results just referred to it was found, as already stated, that the hydrolytic enzyme of the harvested seed was very feeble in one sample and completely inactive in the other. But many seed samples had been examined prior to that time by the auto-enzyme process and had yielded HCN readily. This lack of enzyme power, therefore, seemed to be a property of fresh seed only.

Some samples of known origin and age were therefore procured from the bazaar and the test, which had now become a standard procedure for judging enzyme activity, was applied to these. The results obtained are given in Table XV.

Judging by the method previously employed, in every case the new seed was found to possess weak enzyme power and the old seed strong enzyme power. This enzyme activity has consequently been developed during storage.

TABLE XV.

Enzyme activity of new and old bazaar samples.

		HCN OBTAINED FROM 36 GRM. SEED BY		REMARKS
		Auto-enzyme process	Acid-hydrolysis	
New seed	1 ..	0·0072	0·0090	Enzyme HCN less than acid-hydrolysis, therefore weak enzyme.
	2 ..	0·0066	0·0094	
	3 ..	0·0012	0·0022	
Old seed	4 ..	0·0100	0·0084	Enzyme HCN more than acid-hydrolysis, therefore strong enzyme.
	5 ..	0·0100	0·0050	
	6 ..	0·0146	0·0089	

An experiment to study the effect of storage conditions on enzyme activity was therefore commenced.

For this purpose selected strains of seed were available. A strain having a high HCN content and one having a low HCN content were chosen for the work. The storage conditions tested were as follows :—

1. *Dry air ventilation.* To attain this, seeds were packed into a winchester bottle. Through the bung an air inlet tube was passed to the bottom and an exit tube attached to draw air from the top. 1000 c.c. of air bubbled slowly over H_2SO_4 were daily drawn through all bottles undergoing dry ventilation.

2. *Moist air ventilation.* The procedure was exactly as above with this difference that the air instead of being bubbled through H_2SO_4 was bubbled through water.

3. *Dry air unventilated.* Seeds were packed in desiccators with a beaker containing strong H_2SO_4 imbedded, up to its rim, in the seed.

4. *Moist air unventilated.* Seeds were packed into a winchester bottle, a definite amount of water added and the bottle sealed. The moisture contents of the seeds at the time of bottling was—

Sample B—high HCN content ... 8·375 per cent.
Sample A—low HCN content ... 8·115 per cent.

Sample B was bottled on the 23rd February and Sample A on the 12th March 1920. The table below shows weights of seed used for the experiment:—

TABLE XVI.

Weighings of storage samples.

Sample	Method of storage	Original weight	Final weight	Gain + Loss −	Date of opening
A. Good seed.	Dry ventilated ..	2487·2	2485·1	− 2·1	12–10–20
	Ditto ..	2241·8	2242·5	+ 0·7	9– 8–20
	Moist ventilated ..	2189·4	2187·7	− 1·7	14–10–20
	Ditto ..	2173·5	2175·7	+ 2·2	9– 8–20
	Dry unventilated ..	1145·4	1128·3	− 17·1	16– 8–20
	Ditto ..	1050·0	1028·5	− 21·5	18–10–20
	Moist unventilated ..	2148·4	2210·8	+ 62·4	16– 8–20
	Ditto ..	2189·9	2251·0	+ 61·1	20–10–20
B. Bad seed.	Dry ventilated ..	2444·4	2442·4	− 2·0	12–10–20
	Ditto ..	2225·0	2224·4	— 0·6	11–8–20
	Moist ventilated ..	2414·8	2417·8	+ 3·0	14–10–20
	Ditto ..	2397·9	2400·6	+ 2·7	11– 8–20
	Dry unventilated ..	1153·9	1134·6	− 19·3	18– 8–20
	Ditto ..	1090·9	1067·5	− 23·4	18–10–20
	Moist unventilated ..	2104·6	2166·4	+ 61·8	18– 8–20
	Ditto ..	2197·2	2257·7	+ 60·5	20–10–20

Certain points in these figures have to be noted. In the first place the data show that neither dry nor wet ventilation had any measurable effect on the moisture content of the seed. The vapour tension of the air used in these experiments was not determined. The air was simply bubbled through water in the one case and through H_2SO_4 in the other. To produce measurable changes of moisture content in this seed it is evident that the vapour tension must be made either much higher or much lower than was the case in these tests. The procedure, though it was quite ineffective as regards alteration of moisture content in the seed, was not without effect on the enzyme activity as will be seen later. The respiration of the seeds in these two tests was also examined for a time. No difference was found.

The wetted unventilated seeds had increased their moisture content from the initial 8·2 per cent to 10·7 per cent. This change in moisture content has produced marked effect on enzyme activity.

The seeds submitted to dry unventilated storage were slowly desiccated by the treatment. Their moisture content by the end of the experiment had fallen to 6·4 per cent.

The intention was to examine one sample of each series at intervals of six months. Only one such period was completed. Before the time for the next examination the writer had to leave ; but it was possible to complete a second set of determinations two months after the first.

The samples were examined with the greatest care, auto-enzyme determinations all being carried out in duplicate and the time factor in this process studied. Acid-hydrolysis was done in triplicate. As the results are of considerable significance the data are given in full detail in the accompanying table :—

TABLE XVII.

			Original fresh seed	First sampling				Second sampling			
				Dry ventilated	Moist ventilated	Dry unventilated	Moist unventilated	Dry ventilated	Moist ventilated	Dry unventilated	Moist unventilated
A. Good seed.	Acid hydrolysis		0·0014	0·0008	0·0010	0·0014	0·0020	0·0026	0·0010	0·0014	0·0018
			0·0012	0·0009	0·0010	0·0016	0·0020	0·0024	0·0010	0·0014	0·0018
			..	0·0009	0·0010	..	0·0020	0·0024	..	0·0014	0·0018
	Auto-enzyme.	1 hr.	0·0002	0·0006	0·0006	0·0004	0·0008	0·0006	0·0006	0·0006	0·0012
			..	0·0006	0·0006	0·0004	0·0008	0·0006	0·0006	0·0006	0·0012
		2 hrs.	..	0·0006	0·0006	0·0006	0·0010	0·0014	0·0006	0·0006	0·0014
			..	0·0006	0·0006	0·0006	0·0010	0·0012	0·0006	0·0006	0·0014
		4 hrs.	0·0002	0·0004	0·0006	0·0006	0·0012	0·0016	0·0006	0·0006	0·0014
			..	0·0004	0·0006	0·0006	0·0012	0·0016	0·0006	0·0006	0·0014
		8 hrs.	..	0·0002	0·0006	0·0002	0·0006	0·0010	0·0004	0·0004	0·0012
			..	..	..	..	..	0·0010	0·0004	0·0004	0·0012
		24 hrs.	0·0006	0·0000	0·0006	0·0000	0·0010	0·0006	0·0004	0·0001	0·0012
			..	..	..	..	..	0·0006	0·0004	0·0001	0·0012

TABLE XVII—*contd*

		Original fresh seed	First sampling				Second sampling			
			Dry ventilated	Moist ventilated	Dry unventilated	Dry unventilated	Dry ventilated	Most ventilated	Dry unventilated	Moist unventilated
Acid hydrolysis ..		0·0138	0·0144	0·0146	0·0136	0·0140	0·0130	0·0136	0·0140	0·0134
		0·0140	0·0144	0·0144	0·0134	0·0142	0·0134	0·0140	0·0140	0·0136
		..	..	0·0148	..	..	0·0132	0·0140	0·0138	0·0132
B. Bad seed. Auto-enzyme. 1 hr. ..		0·0016	0·0056	0·0060	0·0054	0·0094	0·0066	0·0065	0·0056	0·0114
		..	0·0056	0·0060	0·0054	0·0094	0·0065	0·0066	0·0058	0·0114
2 hrs. ..		..	0·0060	0·0074	0·0054	0·0094	0·0064	0·0080	0·0057	0·0116
		..	0·0060	0·0074	0·0054	0·0094	0·0066	0·0079	0·0058	0·0116
4 hrs. ..		0·0022	0·0066	0·0074	0·0054	0·0094	0·0066	0·0074	0·0058	0·0116
		..	0·0066	0·0074	0·0054	0·0094	0·0066	0·0074	0·0058	0·0116
8 hrs. ..		..	0·0066	0·0074	0·0052	0·0094	0 0060	0·0074	0·0056	0·0110
		..	..	..	..	..	0·0060	0·0074	..	0 0110
24 hrs. ..		0·0060	0·0066	0·0074	0·0054	0·0094	0·0064	0·0068	0·0058	0·0102
		0·0064	..	..	..	..	0·0064	0·0070	0·0056	0·0106

The results obtained with the second test of dry ventilated sample B are given in full with the other figures. The results from this one bottle cannot be made to fit in with the other figures. They are inexplicable to the writer. From the remaining data certain points stand out quite clearly.

The difference between unventilated dry and wet storage is marked. In the dry storage the liberation of auto-enzyme HCN is restricted whilst in wet storage it is increased.

In comparing dry and wet ventilated storage we have only 3 sets of data to base our conclusions on. The differences produced by the two forms of treatment are slight ; but they are perfectly regular. In every case the moist ventilated sample, compared with the dry ventilated, shows a slightly increased production of HCN by the auto-enzyme process. This it should be remarked has taken place in spite of the fact that the moisture content of the seed has not been altered by an amount that could be measured (see Table XVI). The

result is of interest in showing that altogether imperceptible changes in the moisture conditions of the seed will cause perceptible enzyme changes.

A point which is quite definite also is that in every case after seven months' storage, even under the worst conditions, the acid-hydrolysis invariably yields higher figures than the auto-enzyme process.

Finally, with regard to the auto-enzyme process it is seen that the hydrolysis attains maximum efficiency within one hour, further action generally producing no increase whatever.

To bring out clearly the character of these stored samples one set of typical figures A, A, has been selected and tabulated together with results from typical bazaar seed in the accompanying table :—

TABLE XVIII.

Grm. HCN from 36 *grm. seed.*

	Fresh original A	Dry stored A	Bazaar fresh	Bazaar old	Bazaar fresh	Bazaar old
Acid hydrolysis ..	0·0139	0·0135	Not determined.	Not determined	0·0094	0·0050
Auto-enzyme 1 hr. ..	0·0016	0·0054	0·0070	0·0098	..	..
Auto-enzyme 2 hrs. ..	..	0·0054	0·0086	0·0124	..	..
Auto-enzyme 4 „ ..	0·0022	0·0054	0·0088	0·0124	..	..
Auto-enzyme 8 „ ..	..	0·0052	..	..	..	..
Auto-enzyme 24 „ ..	0·0062	0·0054	0·0096	0·0124	0·0066	0·0100

This table gives the characteristics of normal samples.

It will be seen that the auto-enzyme figures for our fresh and stored samples correspond fairly well with the typical fresh and old bazaar samples respectively. When auto-enzyme and acid-hydrolysis figures are both taken into account our fresh sample behaves normally. The acid-hydrolysis figure, as should be the case with fresh samples, is much higher than the auto-enzyme figure.

Our stored sample which coincided with the old bazaar sample when treated by the auto-enzyme process is quite different when the acid-hydrolysis figure is considered. Old seeds invariably yield a higher figure by auto-enzyme than by acid-hydrolysis, whilst our stored samples give without exception the opposite result.

We may say therefore that according to one test the stored seeds are old, according to the other test they are not old. We may conclude for the present that while they have changed in character to some extent during storage they have not yet advanced to the stage of being called old.

The storage results must also be compared with figures for seeds which were exposed freely to the air in a room by being spread in a single layer on wicker baskets. These seeds gave the following results :—

TABLE XIX.

Analysis of samples stored on trays.

	FEBRUARY		APRIL		JUNE	
	Auto-enzyme	Acid hydrol.	Auto-enzyme	Acid hydrol.	Auto-enzyme	Acid hydrol.
Sample A ..	0·0000	0·0026	0·0007	0·0026	0·0010	0·0010
Sample B ..	0·0016	0·0144	0·0036	0·0085	0·0120	0·0090

These figures show that seed thus freely exposed assumes the normal character of an old seed within four months. Our stored samples were still very far from this stage after six months' storage. The experiments indicate that storage under all the conditions tested restricts vital changes.

It is possible that conditions approaching the storage tests may occur in very large heaps. In any case the conditions existing in such heaps or in sacks could evidently be gauged by examining the seeds and comparing the results with the figures given above.

In small bazaar lots the conditions no doubt approximate to our trays.

The hydrolytic power of stored seeds. Referring again to Table XVII it will be seen that HCN determined by auto-enzyme action is higher after storage than it was in the original fresh seed. This should be an index of increased hydrolytic power.

It has already been observed also that this auto-enzyme liberation of HCN in the stored seeds is completed within one hour. Further action does not increase the yield. This would lead us to the belief that the hydrolytic enzyme is present in a very active form in these seeds.

Turning, however, to the acid-hydrolysis figures we find these are very much higher than the auto-enzyme results. The latter process therefore, although it comes to an end in one hour, is very far indeed from complete. A process which comes to an end in less than one hour and is so far from complete can scarcely be considered a normal enzyme hydrolysis.

Auto-digestion of HCN in the seed. Table XVII shows not only that maximum hydrolysis is attained very quickly but that further enzyme action leads in some cases to actual diminution of the HCN liberated.

We are here dealing with small quantities it is true but the precision with which the figures repeat themselves confirms the indications they give.

From previous work we know what the significance of this decrease is. The seed during the slow-drying process has developed the power of auto-digestion. The test is a very delicate one and may be found useful in future work. The effect of this auto-digestion on the yield of HCN by the enzyme process cannot be lost sight of. It no doubt partly accounts for the fact that the HCN liberated does not increase beyond a certain amount.

We have to conclude that the quantity of HCN liberated is the resultant of a series of balancing processes.

With regard to normal old seed a word must be added. Determinations of hydrolytic power of old seeds were carried out and the seed powder found to contain an active hydrolytic enzyme. Old seeds also invariably yield higher HCN figures by the auto-enzyme process than by acid-hydrolysis.

The above results with stored seed however cast some doubt on the efficiency of the auto-enzyme process with old seed even.

HCN content of seeds. The two cultures of beans used for the storage experiments described above were selected by the writer in 1915 as good and bad respectively. They have been grown regularly every year since then and each culture has maintained its characteristics perfectly.

It should be mentioned that the difference in HCN content is apparent only in the seed. By all the tests which are at present available the plants cannot be distinguished from one another. Both are full of HCN. The low HCN content of the good seed is therefore significant.

The plant has the property of producing seed in which the enzyme activities are so balanced that the HCN content remains slow. Considering the great difference between these two cultures in this respect it does not seem impossible that yet another culture should exist in which the balance is maintained at so low a level that HCN cannot be traced.

SUMMARY.

The chief points brought out during the course of this work may be summarized as follows :—

1. On plunging fresh leaves into boiling water the glucoside is hydrolysed to a considerable extent and part of the HCN is converted into a form which is not recoverable by acid hydrolysis. Similar but not identical effects have been recorded with other plants.

2. *The effect of drying the leaf.* It has been found that there is a fundamental difference between sun-drying and slow-drying. During sun-drying hydrolysis takes place with evolution of HCN. The residual dry leaf when plunged into boiling water evolves further large amounts of free HCN—an effect which fresh leaf does not produce.

   Another peculiarity of this sun-dry leaf is that it continues to evolve free HCN when treated by dry air aspiration. It was shown that this is probably due to a continuance of enzyme action.

   The amount of HCN evolved by sun-drying depends upon the initial enzyme activity of the leaf. Sun-drying directs the vital processes towards liberation of HCN.

   Slow-drying also causes loss of HCN ; but in this case none is evolved, auto-digestion taking the place of evolution.

   The resulting dry tissue is able to destroy HCN as such or HCN as glucoside. The enzymic balance of this slowly dried material approaches that found to occur in fresh tissue of other plants.

3. The hydrolytic power of young leaf is greater than that of old leaf and fresh leaf is somewhat more active than sun-dried leaf.

4. HCN has been found to have a powerful effect on cell permeability in the fresh leaf. It appears therefore that HCN in this plant acts as a regulator or Hormone.

   The presence of HCN has also been shown to bring glucoside hydrolysis to an end prematurely. The effect, which is probably due to paralysis of the enzyme, must play an important part in regulating HCN liberation within the plant.

5. In the fresh green plant the hydrolytic enzyme is also active in the stalk ; but it becomes weak as the plant matures.

6. The green pod and young ripe seed are peculiar in possessing practically no hydrolytic power.

7. The seed is found to develop hydrolytic power as it grows older.

8. The rate of development of hydrolytic activity in the seed depends upon the method of storage. Fresh seeds kept in the open air develop full characteristics of old seed within four months. Samples stored in bottles were still very far from this stage after six months' storage.

9. The drying of seed during storage reveals the power it possesses for auto-digestion of HCN. It is doubtless owing to this property that certain cultures have seeds with low HCN content.

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BY

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Agricultural Chemist, Assam

AGRICULTURAL RESEARCH INSTITUTE, PUSA

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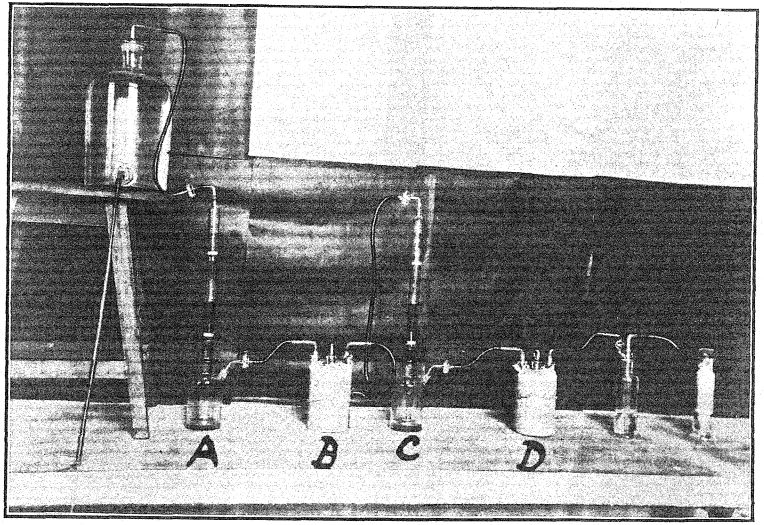

Apparatus used to study Comparative Co_2 evolution from soils.

PLATE I.

STUDIES OF AN ACID SOIL IN ASSAM, II.

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LABORATORY work undertaken in connection with certain field experiments on the Government Farm at Jorhat, Assam, during the past 15 years has revealed what very serious losses of soil nitrogen may take place from the cultivated surface layer of highland areas in a climate like that of Assam. The normal rainfall is of the order of 80 inches, by far the greater portion of which falls within the five months from May to September.

At the time of its acquisition the farm site was a grazing area growing only coarse grasses and low scrub jungle. Representative soil analyses will be found in the Appendix.

Some idea of the nitrogen losses may be gathered from the following :—

Area	Initial N % 0 – 6″	Final N % 0 – 6″	Percentage of initial N lost	Lb. N per acre lost from surface 6″	Remarks
Block C .. Plot IA ..	0·090 (in 1911)	0·055 (in 1921)	39 in 10 years	Some 700 lb. in 10 years	Land cultivated but uncropped and unmanured.
Block E ..	0·115 (in 1909)	0·078 (in 1921)	32 in 12 years	Some 740 lb. in 12 years	Land under sugarcane rotation. Some 750 lb. N have been added in manures in 12 years and 5 green manure crops have been ploughed in.

Similar large losses of nitrogen have been observed on the new alluvium in the Kamrup District of Assam where cultivable waste land was opened out for sugarcane.

For the purpose of obtaining the necessary soil samples a soil auger was used throughout, the number of samples taken per acre varying from 800 in one series of plots to 8,000 per acre in another series. The degree of accuracy thus obtained in sampling is shown by the following analyses of duplicate composite samples taken from four plots :—

Nitrogen percentage on dry soil.

Plot No.	Sample 1	Sample 2
1 ..	0·095	0·094
2 ..	0·090	0·090
3 ..	0·086	0·087
4 ..	0·074	0·075

Again, duplicate analyses of each sample were made by the Kjeldahl method, using 15 grm. soil, every possible refinement being introduced to reduce error alike in the digestion, distillation and final titration. In the result the difference between duplicate analyses of the individual samples very rarely exceeded 1 mg. of nitrogen per 100 grm. soil, and on the average was less than this.

In connection with experiments on the use of lime on this soil which have been in progress for the past 12 years, the figures in Table I are of interest.

It was shown in a previous Memoir (Chem. Ser., III, 9) of the Department of Agriculture in India entitled "Studies of an Acid Soil in Assam" that the primary requirement of this soil for most farm crops was a base of some sort. The use of lime has led to a very great increase in the yield of sugarcane and other crops. The plots from which the figures in Table I are derived form part of an experiment dating from 1911 to test the value of large initial lime dressings versus smaller and more frequent ones in a crop rotation. The four plots in question have not been manured in any way beyond receiving the differential lime treatment specified. The case is therefore not complicated by the question of direct nitrogen additions. The crops taken during the 10 years 1911-1921 include :—

	No. of crops
Aus paddy (*Oryza sativa*).	2
Oats (*Avena sativa*).	4
Jowar (*Andropogon Sorghum*).	5

	No. of crops
Rape (*Brassica campestris*).	2
Barley (*Hordeum vulgare*).	1
Matikalai (*Phaseolus mungo* var. *radiatus*).	1
Maize (*Zea Mays*).	1
Soybean (*Glycine hispida*).	1
Gram (*Cicer arietinum*).	1

Beyond carrying a small paddy crop, plot IA has returned no crop at all; the absence of lime prevents this. The three limed plots have throughout carried crops, naturally small in the absence of other manuring, of all varieties sown. The removals of nitrogen in grain are given in the table, but are only available for the six years 1911–1917.

It is necessary to emphasize here, for a proper understanding of the table, that up to the end of 1919 the plots were regularly cropped twice a year; in 1920 it was decided to let the plots lie fallow, the effect of which on soil nitrogen loss was very marked as will presently appear.

For the period 1911–1919 it will be seen that although from each of the plots Nos. 2A, 3A and 4A at least 100 lb. nitrogen per acre had been removed in grain and straw, nevertheless these three limed plots lost in the eight years less of their initial nitrogen than did the unlimed plot IA, and that the loss was least in the case of plots 3A and 4A which had the heavier initial lime dressings and which carried the heavier crops during the first five years of the experiment. In other words the plots which carried crops lost appreciably less nitrogen than did the one which failed to crop, and the heavier the crops the less the loss. Other work, to be presently described, has proved that limed plots lose a good deal more nitrogen in a given time than do unlimed ones provided both are uncropped. This fact would still further improve the argument in favour of the preventive action of growing crops on soil nitrogen loss as indicated by the above results. This is again emphasized by the effect of the fallow year 1920 referred to above. Thus we find that for the period 1919–1921, embracing the fallow year, the limed plots 2A, 3A and 4A lost a considerably greater amount of nitrogen than did plot IA. So much so that it is true to say that this one fallow year in itself sufficed to negative almost completely, as the figures show, the conservative action of the cropping of the whole previous eight years, the limed plots on the average losing more than three times as much nitrogen during the fallow as the unlimed plot.

The position of these four plots just before the fallow in regard to lime requirement, nitrogen in surface six inches, and relative cropping power as

indicated by a crop of *jowar* taken just previous to the fallow year, is shown below :—

Plot No.	Nitrogen in surface six inches	Lime requirement (Parts CaO per million)	Relative cropping power
	Per cent.		
1A	0·058	690	0
2A	0·061	Nil	100
3A	0·067	280	92
4A	0·066	340	61

It will be seen that in regard to cropping power the nitrogen content of these soils plays a very insignificant part as compared with the lime requirement.

Considered in conjunction with the figures in Table I it appears then that during the fallow period the nitrogen loss varied inversely as the lime requirement.

TABLE I.

Plot	Treatment	Soil nitrogen percentage in 1911 0 – 6″	Soil nitrogen percentage in 1919 0 – 6″	Soil nitrogen percentage in 1921 0 – 6″	Percentage of initial nitrogen lost in eight years 1911–1919	Percentage of initial nitrogen lost in ten years 1911–1921	Nitrogen, lb. per acre, lost from surface six inches for eight years 1911–1919	Nitrogen, lb. per acre, lost from surface six inches for ten years 1911–1921	Nitrogen, lb. per acre, removed in grain for six years 1911–1917	Remarks
1	2	3	4	5	6	7	8	9	10	11
							lb.	lb.	lb.	
1A	No lime	0·090	0·058	0·055	35	39	640	700	4	Cultivated regularly but uncropped as no crops grew in absence of lime.
2A	Lime total 4,800 lb., *i.e.*, 800 lb. annually for six years from 1911–1916 ..	0·085	0·061	0·050	28	41	480	700	70	Cropped regularly twice a year except in 1920 which was a fallow year.
3A	Lime total 4,800 lb., *i.e.*, 2,400 lb. in 1911 and again in 1914	0·091	0·067	0·056	26	38	480	700	80	Ditto
4A	Lime total 4,800 lb., in one initial dressing in 1911..	0·089	0·066	0·058	26	35	460	620	82·5	Ditto

The very marked influence of lime on the rate of nitrification in this soil is well shown in Fig. 1. It will be seen that the nitrate formation in 66 days was in the case of the limed soil twice that in the case of unlimed soil.

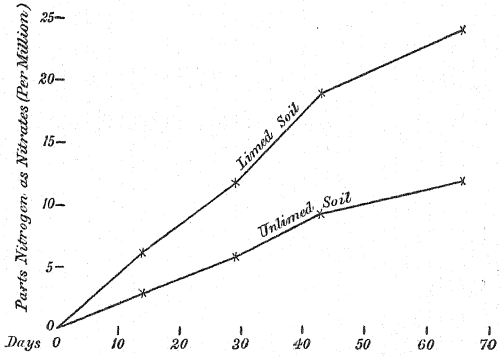

FIG. 1.
Nitrification in limed and unlimed soil.

Both cultures during the incubation period were maintained side by side at the same temperature 28°—30°C. and at a moisture content of 16·7 per cent.

The more rapid loss of soil nitrogen therefore from the limed plots during the fallow may probably be ascribed chiefly to increased loss of nitrates in drainage waters. In the absence of a lysimeter it was attempted to settle this point in another way, taking advantage of the well-known action of sugar in preventing the accumulation of nitrates; and though, owing to the later ascertained fact that this soil assimilates nitrogen from the air asymbiotically in the presence of lime and sugar, the results in regard to the increased loss of nitrogen as nitrates as a result of liming are indefinite, they are sufficiently interesting to describe.

Four small field plots, $\frac{1}{100}$ acre each, were treated as follows :—

Plot 29 E. Untreated.

„ 29 W. Ground limestone to supply 300 per cent. of lime requirement to depth of four inches.

„ 40 E. Ground limestone as above, plus periodical dressings of sugar.

„ 40 W. Ground limestone as above, plus sugar as 40 E, plus sodium phosphate.

Sugar was applied at intervals of a few weeks to plots 40 E and 40 W, as soil and weather conditions permitted, at the rate of 0·045 per cent. of the weight of the surface four inches of soil.

Sodium phosphate was applied at the rate of 25 lb. P_2O_5 per acre on two occasions to plot 40 W.

All plots were cultivated with the Planet Jnr. cultivator frequently and never more than four inches deep, and were uncropped, being kept perfectly clean even of weeds.

The plots were sampled periodically 0—4″ at the rate of 8,000 borings per acre. The surface soil on these plots was shallow but nowhere less than five inches deep, but varied considerably in depth. On this account it was decided to limit cultivation and sampling to a depth of four inches. Sampling to six inches deep would have included a little sub-soil in some cases which would have vitiated the results.

The fall in nitrogen percentage of the surface four inches is shown graphically in Fig. 2 for the period August 1919 to November 1921.

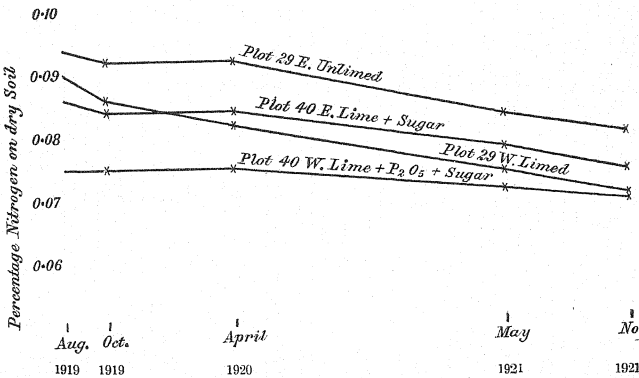

FIG. 2.
Fall in nitrogen of surface 4 inches of soil of variously treated plots.

It will be seen that the plots differed in initial nitrogen percentage and this is unfortunate as it may have influenced the extent of loss in some measure. Perhaps the fairest estimate of relative nitrogen loss will be gained

from a statement of the percentage of initial nitrogen lost for the whole period under review :—

Plot No.	Percentage of initial nitrogen lost
29 E	14·9
29 W	22·2
40 E	13·9
40 W	8·0

The loss in the case of the sugared plots represents the algebraic sum of the actual loss by leaching, etc., and any gain of nitrogen by asymbiotic assimilation.

Briefly it will be seen that, with the exception of the limed plot 29 W, there was no loss of nitrogen during the drier and colder months October to April. At this period the sugar used on the limed plots 40 E and 40 W sufficed to prevent the loss occasioned by lime alone.

For the whole period, lime alone increased the loss considerably over the unlimed plot. This result was reversed and the apparent loss was much reduced when sugar, and more specially sugar and phosphoric acid, was used as well as lime.

On several occasions estimations of nitrate nitrogen were made on the soil samples of the above plots as they came from the field. Qualitative tests for nitrites failed to reveal their presence at any time. Figures are given in Table II.

TABLE II.

Date	Plot	Nitrogen as nitrates. Parts per million	Remarks
August 12, 1919 ..	All four	1·2	Previous to the addition of any thing to the plots. Weather wet.
October 30, 1919 ..	29 E	7·2	Rains just ended. Soil moisture good. Sugar applied to 40 E and W on October 15.
	29 W	9·9	
	40 E	6·0	
	40 W	7·2	
July 1, 1921 ..	29 E	0·4	
	29 W	0·8	Plots 40 E and W last sugared on June 11.
	40 E	2·8	
	40 W	2·8	

TABLE II—*contd.*

Date	Plot	Nitrogen as nitrates. Parts per million	Remarks
July 19, 1921 ..	40 W	0·0	Last sugared on July 12.
July 21, 1921 ..	29 E	0·8	
	29 W	1·2	
August 18, 1921 ..	29 W	2·0	40 E. last sugared on July 12.
	40 E	3·6	
September 6, 1921 ..	40 E	2·4	Last sugared on August 30.
November 7, 1921 ..	40 E	1·2	40 E and W last sugared on November 1. Last rain on October 29.
	40 W	1·2	
	29 E	3·6	
	29 W	4·4	
November 17, 1921 ..	40 E	2·0	Last sugared on November 9.
	40 W	1·2	
November 19, 1921 ..	40 E	Trace	Last sugared on November 17, after sampling on that date. No rain since October 29.
	40 W	Trace	

N.B. Owing to absence on leave during the greater part of 1920, no samples were taken between April 1920 and May 1921. However, sugar was regularly applied to Plots 40 E and W during this period, the plots being kept cultivated and clean.

It will be observed that sugar was too infrequently applied and insufficient in amount to prevent all nitrification in those plots which received it. More specially during the rainy period it would appear that the nitrate determination was in most cases too long delayed after sugaring to note the immediate effect of the latter. With the cessation of the rains the reduction of nitrate by sugar is clear from the determinations made on November 7, 1921, but more specially on November 17 and 19, 1921. In the latter case the sugar added on November 17 almost entirely eliminated all nitrates within two days.

Quite interesting is the apparent fillip given to nitrification after the sugar is removed from the soil, indicated by the figures of July 1, 1921, and August 18, 1921.

This was very strongly marked in laboratory soil cultures in several cases. A good instance of this is shown graphically in Fig. 3. Cultures I and II consisted of equal weights of a sample of limed soil, kept at 16·7 per cent. moisture side by side at the same temperature 28°–30°C. To Culture I

nothing was added, while with Culture II one per cent. of saccharose was mixed. At the end of 14 days sugar was found to be absent by the d-naphthol test

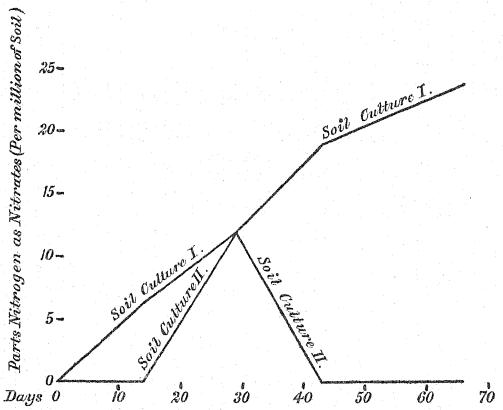

FIG. 3.

in Culture II which at this time showed an entire absence of nitrates, though Culture I contained 6·4 parts nitrate nitrogen per million. No further sugar was used at this point, and at the end of 29 days both cultures showed an identical nitrate nitrogen content of 12 parts per million. Thus in the second period of 15 days Culture II had developed as much nitrate as did Culture I in 29 days. At this point a further addition of sugar one per cent. was made to Culture II. At the end of 14 days it was found that the sugar had destroyed the whole of the nitrates in Culture II, while Culture I had advanced to 19 parts per million. One further addition of sugar was made to Culture II, and at the end of 66 days it still contained no nitrate, while Culture I showed 24 parts per million. It would appear then that sugar not only prevents the accumulation of nitrates but also destroys nitrates already formed, and so long as the merest traces of sugar persist no nitrates can be detected. With the total disappearance of sugar nitrification appears to proceed immediately at a greatly enhanced rate, which suggests either an increased activity of the organisms responsible for nitrification—a theory hard perhaps to reconcile with the immediately preceding severe repression whilst sugar is present—

or the accumulation under the influence of sugar of very readily nitrifiable material. The possibility, I think, cannot be excluded that nitrification does actually take place, and is perhaps stimulated in the presence of sugar, the nitrates being broken down as fast as formed in the presence of excess of sugar. This might serve to explain the observed enhanced rate of nitrification immediately following the final disappearance of the sugar. Nitrites could not be detected in water extracts of these two cultures at any time.

There remains the possibility of the accumulation of salts of ammonia in the presence of sugar. No evidence, however, in favour of this was found. A soil culture which had been sugared periodically over a period of eight months showed no ammonia nitrogen at all. Many other cultures confirmed this.

It was sought to determine, if possible, what happens when already accumulated nitrates are destroyed by addition of sugar, as was proved in Culture II. Attendant circumstances and conditions do not point to denitrification as ordinarily understood. Though some denitrifying bacteria can apparently operate with a limited supply of oxygen, more thorough aeration prevents it. These cultures were thoroughly aerated. The term "denitrification" as ordinarily understood includes (*a*) the reduction of nitrates to nitrites and ammonia and (*b*) reduction of nitrates to nitrites and of these to gaseous nitrogen. As already pointed out no nitrites were detectable by Metaphenylene diamine at any time and no ammonia was formed. To determine what had happened, a sample of soil was taken, the nitrogen as ammonia and by Kjeldahl being separately determined at once in duplicate. To 2,000 grm. of this soil were then added 1·5 grm. potassium nitrate, the whole very thoroughly mixed, and the nitrate nitrogen determined. Sugar at the rate of 1 per cent. was then added, and the mixture was left for a time, tests being made for sugar every few days and the moisture being kept at 15·5 per cent. At the end of 7 days sugar was completely absent, and nitrates had totally disappeared. The mixture was now Kjeldahled again, and the ammonia nitrogen also determined, with the following results, calculated to dry soil :—

Date	Nitrogen by Kjeldahl	Nitrogen as ammonia	Nitrogen as nitrates (parts per million)	Total nitrogen including that of nitrates
	Per cent.	Per cent.		Per cent.
At commencement ..	0·087	0·000	132	0·100
After seven days ..	0·097	0·000	nil	0·097

Thus while some assimilation of nitrogen may have been occasioned by the sugar added, it would appear that the greater part of the nitrate nitrogen reappears in an organic form.

To further test this point and to eliminate any error due to nitrogen fixation, two more cultures were set up as follows :—

Culture A. 1,000 grm. soil.
1 per cent. sugar.
No nitrate added.
Moisture 16 per cent.

Culture B. 1,000 grm. soil.
1 per cent. sugar.
0·75 grm. Pot. nitrate.
Moisture 16 per cent.

If the sugar caused any fixation of nitrogen it might be presumed to go on equally in either case. At the end of 3 days sugar was still present in both cases, but the nitrate had completely disappeared from Culture B. Nitrites were also completely absent.

The results calculated to dry soil were as follows :—

Culture	Date	Nitrogen by Kjeldahl	Nitrogen as ammonia	Nitrogen as nitrates (Parts per million)	Total nitrogen including that of nitrates
		Per cent.	Per cent.		Per cent.
A	Commencement ..	0·048	0·000	0·0	0·048
	After three days..	0·049	0·000	0·0	0·049
B	Commencement ..	0·048	0·000	102	0·058
	After three days..	0·057	0·000	0·0	0·057

Thus allowing for error in the Kjeldahl estimation it would appear that the whole of the nitrate added to Culture B was broken down and appears under the influence of sugar as organic nitrogen. This fact considered along with the entire absence of ammonia and nitrites points to a type of denitrification differing entirely from what is generally understood by this term, and which, excepting for the presence of sugar, takes place under conditions otherwise those usually associated with a rapid nitrification rather than denitrification.

It may here be remarked that certain other cultures in which straw (1 per cent.) was used, instead of sugar, as a medium for supplying the carbohydrate necessary for nitrogen fixation, liberally supplied with carbonate of lime, well aerated, and kept at the optimium moisture content at a temperature of 28°—30°C. showed no nitrification whatever over a period of 6 weeks. Apparently then straw acts in a similar way to sugar in this respect, though without leading to any demonstrable fixation of nitrogen.

Referring back to Fig. 2, it appears that the net loss of soil nitrogen, expressed as a percentage on initial nitrogen, was much reduced on limed field plots uncropped when phosphoric acid in addition to sugar was applied. The very marked effect of phosphoric acid on this soil especially in a basic form was described in a previous Memoir, one theory advanced for its action being an increase in soil oxidation processes.

A good deal of evidence in regard to its stimulation of soil bacterial processes has accumulated. It was constantly observed in nitrogen fixation cultures using sugar, that adding a little phosphoric acid hastened very materially the disappearance of sugar from the soil medium, at the same time by the way increasing the rate of nitrogen fixation. Thus two cultures of the same soil set up on September 6, to both of which 1 per cent. sugar was added (sodium phosphate supplying 0·01 per cent. P_2O_5 being added in one case only) showed that the sugar had totally disappeared 8 days later from the phosphated culture alone, and nitrification had set in. At the end of 7 weeks sugar was still detectable in the unphosphated soil, nitrates being consequently quite absent; while in the other case the nitrate nitrogen had reached over 32 parts per million by this time.

Measuring this increase in the rate of oxidation of sugar by the increase in evolution of carbon dioxide in a given time, by a method to be described later, it was found that the addition of 0·01 per cent. P_2O_5 as sodium phosphate led to an immediate increase of 50 per cent. in carbon dioxide evolved from a sugared soil, as against a similarly sugared but unphosphated culture, while for a period of 8 days the average increase was over 30 per cent.

Again, using soil cultures to which was added 2 per cent. of chopped cowpeas (*Vigna catjang*), instead of sugar, and measuring the evolution over a period of five weeks, it was proved that the addition of 0·01 per cent. of P_2O_5 led to an average increase of 40 per cent. carbon dioxide evolved.

Nitrogen Fixation.

In conjunction with the field work described above, laboratory studies were made of the nitrogen fixing power of this soil under varying conditions.

The plan of the experiments was as follows :—

I. Soil only.
II. Soil+sugar.
III. Soil+lime.
IV. Soil+lime+sugar.
V. Soil+lime+phosphate.
VI. Soil+lime+phosphate+sugar.

The various cultures were carried out side by side at the same temperature, 28°—30°C., and having a moisture content of 16·5—17 per cent. Where sugar was applied it was added at 0, 31 and 45 days. The soil samples for these experiments were taken from three adjoining plots : for Cultures I and II the soil of an unlimed plot was used ; for III and IV that of a plot limed two years previously ; while in the case of Cultures V and VI the plot was limed two years before and had received two dressings of sodium phosphate supplying 0·01 per cent. P_2O_5 on each occasion.

The nitrogen in each sample was estimated at the commencement, and thereafter at the end of 21, 43 and 66 days.

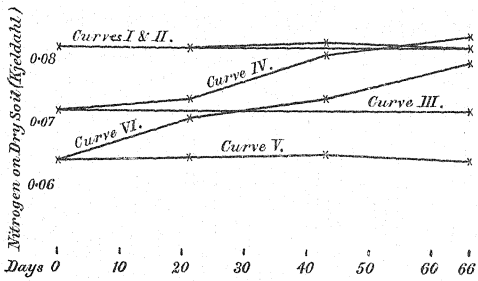

FIG. 4.

Curve I. Soil only — no fixation.
,, II. Soil + sugar — no fixation.
,, III. Soil + lime — no fixation.
,, IV. Soil + lime + sugar — fixation.
,, V. Soil + lime + phosphate — no fixation.
,, VI. Soil + lime + phosphate + sugar — fixation.

The results are shown in Fig. 4 ; briefly the figures show that :—

(1) The addition of lime is necessary with this soil for nitrogen fixation ; no fixation at all took place in the unlimed soil (Curves I and II) even when sugar was used.

(2) Some carbohydrate, *e.g.*, sugar, is required; the limed soil failed to fix any nitrogen in absence of sugar, but did so when sugar was added (Curves III and IV).

(3) Phosphoric acid appears to stimulate fixation; thus the limed and sugared culture (Curve IV) showed a gain of nitrogen in 66 days equal to 16·6 per cent. of its initial nitrogen content, increased where phosphate was used (Curve VI) to 25 per cent.

Culture IV was continued for further observations, as to the effect of the cessation of sugaring, and thereafter to ascertain what limit, if any, there is to nitrogen fixation if sugar is continuously used at intervals. The result is shown in Fig. 5 up to date.

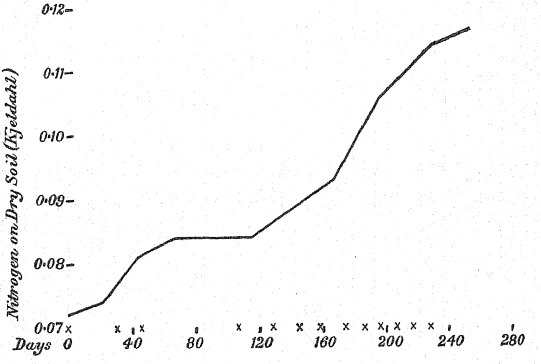

FIG. 5.
Asymbiotic nitrogen fixation.

Sugar was added, 1 per cent. regularly, as soon as it was found to be absent by the d-naphthol test. The points of such addition are indicated by crosses on the abscissa. It will be observed that so long as sugar is supplied, continuous fixation takes place. No sugar was added between the 44th and 106th days, *i.e.*, for two months, and the effect is clearly shown in a temporary cessation of fixation, resumed when sugaring re-commenced. At the time of writing this culture is still fixing nitrogen vigorously, having up to date fixed an amount of atmospheric nitrogen equal to more than 60 per cent. of its original nitrogen content.

The use of sugar as a practical aid to fixation in the field is quite out of the question, and an attempt was made to substitute straw, supplying as it does a large amount of organic matter having a rather small nitrogen content. Paddy and oat straw (dried, finely ground and sieved through 1 mm. sieve) were used in two cultures at the rate of 1 per cent. on the weight of soil, other conditions being made as favourable as possible for fixation. Thus 1 per cent. calcium carbonate was added and sodium phosphate to give 0·01 per cent. P_2O_5. Moisture was maintained at 16·5 to 17 per cent., and the temperature at 28°—30°C. The oat straw contained 0·232 per cent. nitrogen and the paddy straw 0·475 per cent. In 6 weeks no demonstrable fixation was apparent in either case. As previously remarked both these cultures were quite free of nitrates at the end of 6 weeks incubation, which seemed curious in that, excepting for the presence of the straw, all the conditions should have favoured a rapid nitrification. Straw then resembles sugar in preventing the accumulation of nitrates, but unlike the latter appears to be unable, so far as these tests go, to occasion any nitrogen fixation.

The evolution of the CO_2 from soils.

In connection with the foregoing work the evolution of CO_2 from soils of the experimental plots was studied under varying conditions. The behaviour of this soil has been a matter of field experiment and observation for the past 15 years and is well understood, and it was sought to determine amongst other things how far CO_2 evolution is a positive index of improved fertility. The method finally adopted will be readily understood from the photograph appearing as the frontispiece. A sample of the soil under experiment was thoroughly mixed, and 500 grm. placed in each of the Woulff's Bottles B and D. The aspiration was conducted in tandem to ensure equal conditions, the CO_2 evolved in B passing into Reiset A containing baryta, that from D being absorbed in Reiset C. Preliminary experiments using extra intermediate Reisets proved that CO_2 absorption was absolutely complete, under the conditions of the experiment, in Reisets A and C, no CO_2 passing over from Reiset C into Woulff's bottle B for instance.

Previous to introducing any differential treatment several aspirations were made until an equal CO_2 evolution was established in both Woulff's bottles. This done, both bottles were now emptied, and the desired differential treatment introduced in one case only. The soils were refilled into their respective bottles, and aspirations contained. Or, in case it was desired for instance to test two forms of phosphate against each other, after establishing

the preliminary equality in CO_2 evolution, one phosphatic manure was introduced into one bottle, and the other into the second one.

As previously remarked, it was observed that the addition of a little sodium phosphate to a sugared soil hastened the disappearance of the sugar very materially, incidentally increasing the rate of nitrogen fixation also, and presumably the oxidation of any organic matter would be similarly accelerated. To test this, to a given soil 2 per cent. of its weight of chopped green cowpea plants was added, and the moisture content adjusted to 16·5 per cent. Five hundred grammes of the mixture were introduced into each Woulff's bottle and aspirations made until CO_2 evolution was equalized. In one case there was then added 0·01 per cent. of phosphoric acid as sodium phosphate and the aspirations continued with the following results :—

Date	Litres of air aspirated	Duration of aspiration	Milligrams of CO_2 evolved	
			Bottle I	Bottle II
27–9–21	5	About 5 hours	66·0	63·0
28–9–21	5	„	55·0	54·0
29–9–21	Sodium phosphate added to Bottle I.			
30–9–21	5	About 5 hours	68·0	50·0
1–10–21	5	„	50·0	38·0
3–10–21	5	„	65·0	50·0
19–10–21	5	„	11·5	4·0
2–11–21	5	„	9·0	3·7

(*N.B.*—From 3-10-21 to 19-10-21 and also from 20-10-21 to 29-10-21 both Woulff's bottles stood with side tubes open, and were exhausted of any CO_2 daily by the bellows.)

It will be seen that the added phosphate increased the CO_2 evolution by 40 per cent. on the average of five aspirations continued over a period of a month.

The effect of phosphoric acid in the field has always been a marked one in its basic forms. Basic slag for instance influences cropping to a much greater extent than was superphosphate, the latter, while effective if used along with lime, being of little use without it.

The following results were obtained in a test made of CO_2 evolution using basic slag against superphosphate.

Date	Litres of air aspirated	Duration of aspiration	Milligrams of CO_2 evolved	
			Bottle I	Bottle II
27-6-22	5	About 5 hours	34	34
1-7-22	5	„	25	25
4-7-22	To Bottle I was added 0·02 per cent. P_2O_5 as basic slag.			
	„ „ II „ „ 0·02 „ „ „ superphosphate			
7-7-22	5	About 5 hours	34	28
11-7-22	5	„	47	30
14-7-22	5	„	30	19

Here then the much greater field effect of basic slag is accompanied by an increased CO_2 evolution.

So far then the above, amongst other tests, appeared to indicate that greater fertility and increase in CO_2 formation go hand in hand. That this is not invariably so is proved by a further test using sulphate of ammonia. In the field, in absence of preliminary liming, sulphate of ammonia has shown itself to be very toxic to a variety of cropping over a period of years. A small plot to which small annual doses of this fertilizer are added has steadily become more toxic until to-day it is incapable of carrying even weeds in the rainy season, when otherwise weeds are such a terrible nuisance on this farm.

The aspiration results with sulphate of ammonia, using unlimed soil, were as follows :—

Date	Litres of air aspirated	Duration of aspiration	Milligrams of CO_2 evolved	
			Bottle I	Bottle II
18-7-22	5	About 5 hours	34	34
21-7-22	5	„	22	22
22-7-22	To Bottle I added 0·25 grm. sulphate of ammonia (=100 parts nitrogen per million of soil).			
25-7-22	5	About 5 hours	24	20
28-7-22	5	„	23	20
1-8-22	5	„	25	23
18-8-22	5	„	13	12

This result appears to indicate that for sour soils at any rate increase in CO_2 evolution is not invariably correlated with enhanced fertility. Estimations of nitrogen as nitrate made at the conclusion of the aspirations, 36 days from the commencement, showed that the soil to which was added the sulphate of ammonia contained 13·6 parts per million, while the untreated soil had 9·6 parts. Thus of the ammonia nitrogen added to Bottle I, only 4 per cent. had been nitrified in 36 days under optimum moisture conditions and temperature 28°—30°C., a much smaller amount than should be nitrified in the time and under the conditions in the case of a fertile soil.

When one considers the numberless millions of bacteria at work in a soil breaking down organic matter, etc., the various processes being carried on by distinct species requiring different conditions for their best results, it is not hard to realize that under some conditions a species or number of species of bacteria not closely associated with the processes usually considered as vital, *e.g.*, nitrification, fixation of nitrogen and so on, may swamp the more beneficial organisms. When the nitrogen content of a soil passes a certain limit the decay bacteria increase rapidly and in the struggle for existence may easily suppress other species. The use of sulphate of ammonia might conceivably lead to such a state of affairs, and would be accompanied by increased CO_2 evolution. Again, since in certain soil processes, *e.g.*, nitrification, the action of many species is more or less supplementary, one can conceive a definite optimum balance between these various organisms, and anything tending to upset this balance may limit their activities. In view of the fact that the different organisms vary in their requirements, *e.g.*, for air and moisture amount and kind of food, temperature, etc., etc., and also that they are affected unequally by changing environmental conditions, one must conclude that the bacterial equilibrium varies constantly more or less. The use of sulphate of ammonia for instance in our sour soil, where the nitrifying bacteria are already more or less embarrassed by acidity, might further upset the balance of organisms concerned in the nitrification cycle by unduly stimulating the decay bacteria. This would lead to an increased evolution of carbon dioxide and to a limitation of nitrification.

Though a great deal more work along these lines would be necessary before one could attempt to generalize, it would appear that this method of study may prove useful but that more reliable information would be likely to accrue if in all cases before aspiration, and again after aspirations are concluded, estimations of nitrate nitrogen are made.

As a method of estimating comparative CO_2 evolution, the method described leaves little to be desired and is capable of great accuracy.

General.

The work reviewed in this paper proves what very great losses of soil nitrogen may take place in a humid and hot climate with a heavy rainfall during about half the year. On uncropped plots this rate of loss is much increased by liming, and bare fallows in the rainy season are especially wasteful; on the other hand, when cropped, the increase in vegetation due to liming tends to conserve the nitrogen. It seems probable that these losses are incurred chiefly by leaching of nitrates in drainage waters. Although the production of nitrates takes place very slowly in temperate climates, under our conditions it is relatively a much more active process, so much so that probably even on unlimed areas the loss is normally almost entirely due to leaching of nitrates.

When the land is cropped this leaching is reduced naturally, and moreover nitrification may be retarded at times by a reduction of soil moisture as a result of active transpiration. This combined with the nitrogen returned to the soil in the roots and other residues of crops may serve to explain the observed lower rate of soil nitrogen loss from cropped land. The work described provides another reason, if one were necessary, for green-cropping during the rainy season wherever this can be introduced even between standing crops when grown in lines so that the green-crop can be hoed in. By so doing not only is a good deal of waste of soil nitrogen prevented, but the crop itself adds further nitrogen gathered from the air. But apart from green-cropping, even a loose covering of grass and weeds periodically hoed in during the rains must have a conservative effect.

In regard to nitrogen fixation it appears to be absolutely essential that the soil should be limed; phosphoric acid used periodically would seem also to be helpful. As a source of energy for the fixing bacteria sugar is pre-eminent, but, of course, out of the question for field use. Though cellulose decomposition products may serve as the necessary source of energy, so far as my experiments went the use of straw occasioned no fixation. Possibly the decomposition did not proceed sufficiently far or in the right direction under laboratory culture conditions. The conditions obtaining in the field where green-manure crops are ploughed in occasionally, and cowdung is used, and where moisture and temperature conditions vary considerably from time to time, might well result in the production of different decomposition products from those resulting in laboratory cultures.

The nitrogen position on the sugarcane rotation areas on the Jorhat Farm may perhaps have some significance in the light of the work described.

These areas have been under a four-year rotation for the past twelve years or more, embodying two years under cane (manured) and two under green manures and other cropping. They are limed every fourth year, and part of each block also receives a dressing of phosphate once in four years. At the present time the position is this: that in three cases out of four, the phosphated areas show a present average soil nitrogen content some 13 per cent. in excess of that of the otherwise similarly treated but unphosphated areas, equal to an excess of 180 lb. nitrogen per acre 0—6″. It is only fair to state that these phosphated areas have regularly returned bigger leguminous green crops in the rotation, but to balance this, other crops, *viz.*, sugarcane and oats, have removed more nitrogen year by year from these areas. It would need a much greater increase in green crops than has actually resulted to account for the whole of the present excess of nitrogen in the surface soil of the areas receiving phosphatic manure. While it cannot be claimed that this excess is due to asymbiotic nitrogen fixation, the possibility of this being the case certainly cannot be ruled out under the conditions.

Soil work both at Jorhat and in the newly opened out sugarcane tract in the Kamrup District of Assam serves to show that on opening out cultivable waste lands a more or less rapid fall of nitrogen initially must be expected. The mere cultivation of such soils which may have lain as they are for generations under jungle, adding year by year to their stores of humus, must assuredly set up a train of bacterial processes which would liberate in one form or another a good deal of nitrogen. So far there would appear to be no royal road to prevent this, but sound agricultural practice will undoubtedly prevent this process being carried too far. Apart from the use of excessive quantities of nitrogen in manure, under our conditions the adoption of a sound rotation embodying green manuring with the periodical use of lime and phosphate would appear to offer the only practical solution of the nitrogen problem. When these conditions obtain, the apparently inevitable initial fall in soil nitrogen need not occasion excessive alarm. The cane blocks on the Jorhat Farm, though now showing on the average a soil nitrogen percentage amounting to less than 70 per cent. of their initial content in spite of green-cropping and heavy direct manuring for the cane crops, produce to-day from two to three times the crops of sugarcane which it was possible to obtain when the land was newly opened out, and with less direct application of nitrogen in manures than was the case formerly

APPENDIX.

A. Chemical analysis of the Jorhat surface and sub-soils.

		Surface soil	Sub-soil
		Per cent.	Per cent.
Soluble in 26 per cent. hydrochloric acid with 12 hrs. digestion at 100°C	Phosphoric acid (P_2O_5) ..	0·025	0·020
	Potash (K_2O) ..	0·115	0·135
	Lime (CaO) ..	0·154	0·144
	Magnesia MgO) ..	0·166	0·148
Soluble in 1 per cent. citric acid with seven days digestion.	Phosphoric acid ..	0·008	0·008
	Potash ..	0·007	0·011
Loss on ignition=organic matter + combined water .		3·260	1·840
Nitrogen		0·114	0·051
Carbonic acid (CO_2)		0·009	0·007
=Calcium carbonate		0·020	0·015
Reaction		Acid	Acid

B. Mechanical analysis (ignited fractions).

		Surface soil	Sub-soil
		Per cent.	Per cent.
Fine gravel	(3 — 1 mm. diam.)	nil	nil
Coarse sand	(1 — 0·2 mm. diam.)	7·2	6·3
Fine „	(0·2 — 0·04 mm. diam.)	52·5	52·8
Coarse silt	(0·04 — 0·01 mm. diam.)	22·6	17·6
Fine „	(0·01 — 0·002 mm. diam.)	5·0	9·4
Clay	(0·002 mm. diam.)	6·6	9·9
Moisture + Loss on ignition	.. ..	5·1	3·1
		99·0	99·1

C. Moisture constants.

	Hygroscopic capacity	Maximum water-saturation capacity		Minimum water-saturation capacity		Moisture in air-dry soil
		Per cent. of water in saturated soil by weight	Per cent. of water in saturated soil by volume	Per cent. of water by weight	Per cent. of water by volume	
Surface soil..	3·10	31·9	50·5	11·2	13·9	1·30
Sub-soil ..	..	30·0	50·0	7·2	9·1	1·19

D. Acidity.

Expressed as parts quicklime (CaO) required to neutralize one million parts of air-dry soil, the acidity of the surface and sub-soils on the Jorhat Farm varies between the following limits :—

Surface soil 880—1350 parts CaO per million.

Sub-soil 650—770 parts CaO per million.

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MEMOIRS OF THE DEPARTMENT OF AGRICULTURE IN INDIA

A PRELIMINARY NOTE ON THE DECOMPOSITION OF CALCIUM CYANAMIDE IN SOUTH INDIAN SOILS

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A PRELIMINARY NOTE ON THE DECOMPOSITION OF CALCIUM CYANAMIDE IN SOUTH INDIAN SOILS.*

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CALCIUM CYANAMIDE or nitrolim is a nitrogenous fertilizer containing about 20 per cent. of nitrogen derived from the atmosphere.

In Europe, where great developments have taken place in its manufacture, this manure is in common use and has given results very little inferior to ammonium sulphate.

In South India, on the other hand, where a number of trials have been carried out in recent years, the results have been disappointingly erratic. In view of the probable development of hydro-electric power schemes in this country which should render possible the manufacture of the fertilizer at a reasonable cost, it has seemed desirable to re-examine the whole question both in the laboratory and by means of field trials. The work is still very incomplete, but some of the preliminary results obtained may be described with advantage.

Meantime we are continuing the investigation paying particular attention to the factors controlling the formation and decomposition of dicyanodiamide and the differential action of the latter on the nitrification of organic manures as opposed to mineral manures of which we have obtained some evidence. The residual effects, if any, of cyanamide will also be examined.

* Paper read before the Indian Science Congress, January 1923.

Balakrishnamurthi[1] has summarized the results of field trials on the various Government Farms in Madras between the years 1916 and 1920. This summary clearly shows the erratic nature of the results. In some cases the manured plots gave yields lower than the controls, while in others, where an increase was shown, this was so small as to be within the experimental error, or at any rate insufficient to cover the cost of the manure. Many of these experiments, however, were unsatisfactory having been carried out in single or duplicate plots only and hence, as Balakrishnamurthi points out, the results cannot be very seriously entertained.

Field trials.

1919-1920 *and* 1921-1922. The results of experiments conducted on the Central Farm, Coimbatore, during the years 1919-1920 and 1921-1922 with *tenai* (*Setaria italica*), *ragi* (*Eleusine coracana*) and paddy (*Oryza sativa*) on red, garden and wet lands respectively are given in Tables I to IV.

Table I.

Dry red soil. Crop *tenai* (*Setaria italica*).

		No. of plot	Area in cents	Yield in pounds	Acre yield in pounds
Manured		1	5	38	760
		3	5	42	840
		5	5	46	920
		7	5	46	920
		9	5	38	760
	Total ..	..	..	210	4,200
Unmanured . ..		2	5	36	720
		4	5	28	560
		6	5	36	720
		8	5	37	740
		10	5	38	760
	Total ..	..	..	175	3,500
Average ..	Manured ..	..	..	..	840
	Unmanured ..	..	..	..	700
					.. per cent.
Percentage increase		..	..	..	20
Probable error of individual plots		..	..	..	± 9·10
Probable error comparing two sets of five plots each		..	..	..	± 5·74

[1] Balakrishnamurthi. *Year Book, Dept. of Agri., Madras*, 1920-21.

This field was prepared in August, and 10 five-cent experimental plots (300 links $\times 16\frac{2}{3}$ links) were laid out across the field. Nitrolim was applied to alternate plots at the rate of 1 cwt. per acre on 4th August, 1919, and was worked into the soil by the Junior hoe. After the application of the manure a drought intervened so that the sowing of seed was delayed until 15th September, 1919. The germination in all plots was satisfactory, but from the beginning the manured plots showed the more vigorous growth and this continued till harvest time. The amount of rainfall received for the crops was 15·29 inches and this was fairly distributed throughout the period the crop was on the land.

TABLE II.

Red soil. Irrigated *ragi* (*Eleusine coracana*).

	No. of plot	Area in cents	Yield in pounds	Acre yield in pounds
Manured	1	5	123	2,460
	3	5	148	2,960
	5	5	152	3,040
	7	5	144	2,880
	9	5	141	2,820
TOTAL		..	708	14,160
Unmanured	2	5	132	2,640
	4	5	133	2,660
	6	5	143	2,860
	8	5	130	2,600
	10	5	144	2,880
TOTAL	..	..	682	13,640

Average yield	Manured	2,832
	Unmanured	2,728
		per cent.
Percentage increase		3·80
Probable error of individual plots		± 4·36
Probable error comparing two sets of five plots each		± 2·75

The preliminary cultivation was completed on 21st June, 1919, and 10 five-cent experimental plots (400 links × 12½ links) were prepared. The experimental plots were laid out in beds and the manures applied to alternate plots at the rate of 1 cwt. per acre on 23rd June, 1919. Irrigation was given and the *ragi* (seedlings) plants which had previously been raised in a nursery in F. No. 19 were planted in puddle on the 29th June, 1919. The crop received two hoeings and 8 irrigations in addition to rainfall of 4·8 inches. The crop grew well throughout but except that the manured plots remained a darker green there was no other visual difference.

TABLE III.

Wet land. Paddy (*Oryza sativa*). 1919-1920.

	No. of plot	Area in cents	Yield in pounds	Acre yield in pounds
Manured	1	5	231	4,620
	3	5	224	4,480
	5	5	224	4,480
	7	5	224	4,480
	9	5	258	5,160
TOTAL	..	..	1,161	23,220
Unmanured	2	5	200	4,000
	4	5	212	4,240
	6	5	206	4,120
	8	5	163	3,260
	10	5	164	3,280
TOTAL	..	..	945	18,900

Average	Manured	4,644
	Unmanured	3,780
		per cent.
Percentage increase		22·70
Probable error of individual plots		± 9·36
Probable error comparing two sets of five plots each		± 5·90

Puddling operations were completed on 2nd September, 1919, thereafter 10 five-cent plots were laid out and nitrolim applied to alternate plots at the rate of 1 cwt. per acre. Transplanting commenced on 4th September, 1919, with seedlings taken from the nursery in C Block. The seedlings at the time of planting were especially good, being robust and vigorous. All plots grew very well and a slight difference was noticed in favour of the manured plots. The season was a particularly favourable one for paddy and the crop which was harvested on 30th December, 1919, yielded as shown below :—

TABLE IV.

Wet land. Paddy (*Oryza sativa*). 1921-1922.

	No. of plot	Area in cents	YIELD PER PLOT IN LB.		YIELD PER ACRE IN LB.	
			Grain	Straw	Grain	Straw
Unmanured ..	1	5	155	196	3,100	3,920
	3	5	134	200	2,680	4,000
	5	5	156	236	3,120	4,720
	7	5	140	168	2,800	3,360
Manured	2	5	164	224	3,280	4,480
	4	5	138	228	2,760	4,560
	6	5	146	144	2,920	2,880
	8	5	134	148	2,680	2,960
Average for unmanured ..	..	..	146	200	2,925	4,000
Average for manured	..	..	145	186	2,910	3,720

These results are again as erratic as those summarized by Balakrishnamurthi. They have, however, not been so unfavourable as those independently conducted by the Superintendent, Central Farm, Coimbatore, during the previous year when the unmanured plots gave higher yields than the calcium cyanamide plots. The figures of 1919-1920 showed varied increases in favour of nitrolim, and while they were not sufficiently high, except in the case of

paddy, to claim any definite result in favour of the manure, it was considered that the case was sufficiently interesting to warrant further investigation.

In one or two cases the failure of the manure to bring about any marked increase of crop may be attributed to the fact that the experiments were conducted on land already containing a fairly high percentage of nitrogen, while in others a deficiency of phosphoric acid may have been a limiting cause. It is obvious, of course, that these two factors demand careful consideration in laying down plots for any manurial experiment.

In those cases where the application of nitrolim has definitely depressed the yield, this effect has usually been attributed to the formation of dicyanodiamide, a substance believed to have toxic powers.

It was obviously desirable to deal with this latter point first and to ascertain, if possible, the fate of nitrolim in the soil under the conditions prevailing in this country.

A considerable amount of work has been done in Europe and America since nitrolim was first put on the market. Aso[1], Loew[2], Ulpiani[3], Kappen[4], Brioux[5] and Russell[6] are among the earliest investigators who paid considerable attention to the subject, while later contributions have been made by Cowie[7] of the Rothamsted Experiment Station. Cowie gives a brief summary of the earlier work and the results of his own investigations in regard to the decomposition of calcium cyanamide in the Rothamsted and Woburn soils.

Cowie's conclusions were as follows :—

1. Cyanamide readily breaks down in the soil yielding ammonia, which then nitrifies in the usual way. The conversion of cyanamide nitrogen into nitrate is practically quantitative and its effectiveness as a fertilizer is approximately equal to that of ammonium sulphate.

2. Dicyanodiamide has given no evidence of nitrification in the soil even after several months. On the contrary, it is actually toxic to plants, although

[1] Aso. *Jour. College Agri.*, Tokyo, **1**, 1909—1913, 193.
[2] Loew. *Chem. Zeit.*, 1908, No. 57 ; 1909, No. 3.
[3] Ulpiani. *Cent. für. Bakt.* Abt. II, Bd. **18**, 1907, 55.
[4] Kappen. *Cent. für. Bakt.* Abt. II, Bd. **22**, 1909.
[5] Brioux. *Annales de la Science Agronomique*, III, Série 5, Année 1910, Tome I, 280.
[6] Russell. *Jour. Agri. Sci.*, VI, 53.
[7] Cowie. *Jour. Agri. Sci.*, IX, 113.

in small amounts it causes no appreciable injury. It does not affect germination at any of the concentrations used.

3. Dicyanodiamide is also toxic to the nitrifying organisms and stops the normal oxidation of ammonia in soils containing ammonium sulphate. It likewise inhibits the transformation into nitrate of the ammonia produced from cyanamide in the soil and causes an accumulation of ammonia under these conditions. It does not sensibly retard the formation of ammonia from cyanamide.

4. Dicyanodiamide does not appear to affect so drastically the other organisms of the soil, especially those concerned in the decomposition of protein. It exerts little influence upon the numbers developing on gelatine plate or the rate and extent of the decomposition of dried blood.

It will thus be seen that Cowie's work refers largely to the ammonification and nitrification of cyanamide in the soil and the influence of dicyanodiamide on these processes. He has not produced definite evidence in regard to the formation of dicyanodiamide in the soil, but conducted his experiments with dicyanodiamide prepared in the laboratory on the assumption that this was produced in the soil. It is of importance to ascertain whether this assumption is justified and if it be correct that dicyanodiamide is produced, to discover, if possible, the conditions which lead to its formation. Moreover, it seemed unjustifiable to assume that the decomposition of nitrolim in the soil, which can obviously take different courses, would necessarily be the same under the conditions prevailing in South India as was found to be the case in English soils. The difference in quality and texture of the soils, in agricultural practices, in climate and in soil temperatures between the soils of the two countries are so great that, in the absence of any information in regard to the decomposition of nitrolim in Indian soils, we considered it advisable to undertake a re-examination of the subject from the point of view of South Indian agriculture.

The general impressions prevalant in South India in regard to the use of calcium cyanamide are :—

1. That a certain interval of time is necessary between the application of manure and sowing.
2. The crop is liable to suffer owing to the formation of some toxic substance (dicyanodiamide) in the soil.
3. The nitrogen becomes available somewhat suddenly at a time when the crop is not able to utilize it to the greatest advantage.

In order to obtain definite information on the above points, the scope of enquiry, in its early stages, was limited to the following :—

1. What interval, if any, is necessary between the application of the manure and sowing or transplanting ?
2. What is the nature of the decomposition of calcium cyanamide in the soil ?
3. What are the products of decomposition and how quickly are these formed ?
4. Is dicyanodiamide formed at all in the soil, and, if formed, what is its ultimate fate ? Is it injurious and if so how ?

Interval between sowing and the application of manure.

Pot experiments and field trials were instituted to ascertain if any interval is necessary between manuring and sowing. In field No. 44 (Central Farm) a series of five-cent plots were laid out. In each series one plot was left unmanured as control and to four other plots calcium cyanamide at the rate of 1 cwt. per acre (20 lb. N per acre) was applied 3 weeks, 2 weeks, 1 week and 1 day previous to transplanting *ragi* (*Eleusine coracana*). There were thus 5 plots in each group and four groups or 20 plots in all. With the soil from the same field pot culture experiments on similar lines were started, the amount of nitrogen in this case, however, being about $2\frac{1}{2}$ times the amount used in the field. The results of these pot experiments and field trials are given in Tables V and VI and also shown photographically in Plate I, figs. 1–3. Similar pot experiments with paddy were also conducted, the results being given in Table VII.

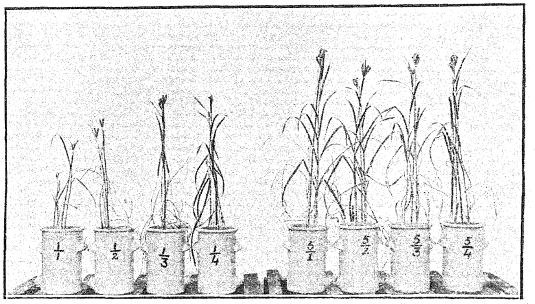

Fig. 1. No manure.

Manured 1 day before planting.

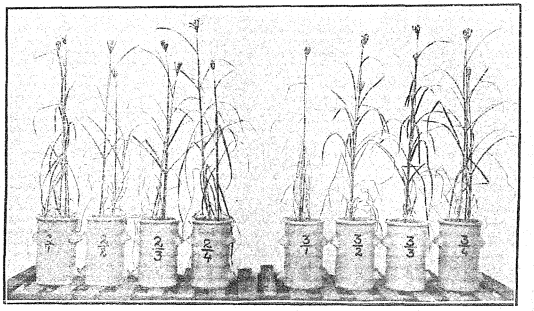

Fig. 2. Manured 3 weeks before planting.

Manured 2 weeks before planting.

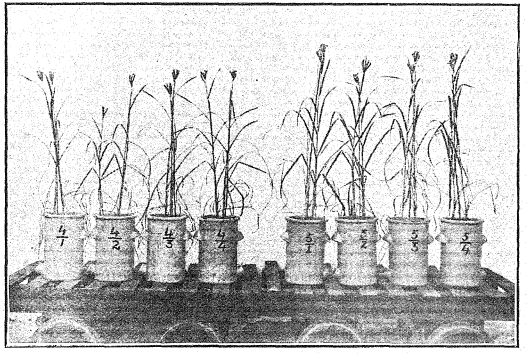

Fig. 3. Manured 1 week before planting.

Manured 1 day before planting.

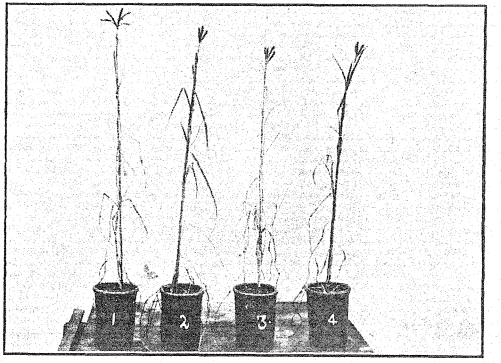

Fig. 4. Large doses of dicyanodiamide applied six months previously.

POT EXPERIMENTS WITH RAGI.

(In Figs. 1—3 100 mg. cyanamide nitrogen per kilo soil.)

PLATE I.

TABLE V.

(Garden lands. Irrigated *ragi*.)

Showing yields of ragi (grain and straw). 1 cwt. nitrolim per acre applied at different times.

No. of series	NO MANURE				MANURE APPLIED 3 WEEKS BEFORE PLANTING				MANURE APPLIED 2 WEEKS BEFORE PLANTING				MANURE APPLIED 1 WEEK BEFORE PLANTING				MANURE APPLIED A DAY BEFORE PLANTING			
	Per plot		Per acre		Per plot		Per acre		Per plot		Per acre		Per plot		Per acre		Per plot		Per acre	
	Grain	Straw	Grain	Straw	Grain	Straw	Grain	Straw	Grain	Straw	Grain	Straw	Grain	Straw	Grain	Straw	Grain	Straw	Grain	Straw
	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.
1 ..	101	184	2,037	3,680	99	197	1,988	3,840	103	175	2,066	3,500	104	198	2,089	3,960	105	207	2,102	4,140
2 ..	114	204	2,286	4,080	115	217	2,306	4 340	106	204	2,139	4,080	115	205	2,317	4,100	119¾	224	2,395	4,480
3 ..	115	211	2,307	4,220	116	224	2,333	4,480	105	205	2,115	4,100	112	219	2 244	4,380	112½	220	2,250	4,400
4 ..	114	202	2,282	4.040	116	204	2,321	4,080	120	216	2,409	4,320	117	208	2,354	4,160	107	179	2.146	3,580
TOTAL ..	444	801	8,912	16,020	446	842	8,948	16,740	434	800	8,729	16,000	448	830	9.004	16,600	444	830	8 893	16,600
AVERAGE ..	111	200	2,228	4,005	111½	210½	2,237	4,185	109	200	2,182	4,000	112	207½	2 251	4 150	111	207½	2.223	4,150

Averages.

	PER PLOT		PER ACRE	
	Grain	Straw	Grain	Straw
No manure	111	200	2,228	4,005
3 weeks before planting ..	111½	210½	2,237	4,185
2 weeks before planting ..	109	200	2,182	4 000
1 week „ ..	112	207½	2,251	4,150
1 day „ ..	111	207½	2,223	4,150

TABLE VI.

Showing results of pot experiments with ragi (Eleusine coracana), 1921.

(Manure applied at 100 milligrammes cyanamide N per kilogramme of soil.)

No. of series	Particulars of manure	Yield	
		Grain	Straw
		Grm.	Grm.
I	No manure	6·45	15·35
II	Manure applied 3 weeks before planting	16·57	31·98
III	,, ,, 2 ,, ,, ,, ...	14·00	28·25
IV	,, ,, 1 week ,, ,, ...	13·07	21·58
V	,, ,, 1 day ,, ,, ...	16·07	30·98

TABLE VII.

Showing results of pot experiments with paddy (Oryza sativa), 1921.

(Manure applied at 100 milligrammes cyanamide N per kilogramme of soil.)

No. of series	Particulars of manure	Yield	
		Grain	Straw
		Grm.	Grm.
I	No manure	38·0	50·0
II	Manure applied 1 week before transplanting and immediately puddled	79·0	105·0
III	Manure applied 1 week before transplanting but not immediately puddled	93·0	110·0
IV	Manure applied a day before transplanting ..	76·0	111·0

The results clearly indicate that in these experiments the interval between manuring and transplanting had no influence on the resulting crop. In the field trials "no manure" plots have given the same results as the manured ones, while in pot experiments, on the other hand, the yield in the case of manured plots is nearly three times that obtained from controls. It was particularly

unfortunate that the land on which the field trials had to be made already contained a somewhat high percentage of nitrogen (0·12 per cent.). The amount of nitrogen added (20 lb. per acre) under these circumstances was perhaps hardly felt. In the case of the pot experiments where the application of nitrogen was much heavier, the results were much more favourable.

In this instance, however, it is sufficient to observe that no harmful influence of any sort was produced even when the interval between the application of the manure and transplanting was reduced to a minimum, either in field or in pot culture.

Decomposition of nitrolim in the soil.

When it is sought to follow the course of decomposition in the soil, serious difficulties are at once encountered. The existing methods of estimation of the substance likely to be produced are in several cases lacking in accuracy, especially in the presence of the other decomposition products. The methods of analysis have, therefore, had to be tested and standardized to suit our conditions. As a preliminary step it was decided to follow the course of decomposition determining (1) dicyanodiamide, (2) urea, (3) ammonia, (4) nitrite and (5) nitrate.

After numerous trials, urea, ammonia, nitrites and nitrates were determined by the methods employed by Cowie, with the exception that in determining urea the urease of red gram (*Cajanus indicus*) was used instead of soya bean urease which is not easily available in South India. Preliminary experiments had shown that *Cajanus* urease[1] was equally specific in action and gave results as satisfactory as those obtained with soya bean urease.

Dicyanodiamide is usually determined by Caro's method as modified by Brioux. This method consists in precipitating a solution containing calcium cyanamide and its decomposition products with silver nitrate in presence of caustic potash, collecting the precipitate and determining the nitrogen by Kjeldahl method. Both in the original Caro and the modified Brioux method, however, about 25 per cent. of the urea present is also precipitated along with the dicyanodiamide.

Harger[2] proposed a new method which depends on the fact that when a solution of silver picrate is added to a solution of dicyanodiamide, the latter is quantitatively precipitated as a double compound of silver picrate and dicyanodiamide. Neither cyanamide nor urea gives any precipitate when their solutions are treated with silver picrate, nor does their presence interfere with the

[1] Viswanath. *Agri. Jour. India*, Special Indian Science Congress Number, 1917.
[2] Harger. *Jour. Indus. Engineering Chem.*, **12**, 1107.

precipitation of dicyanodiamide. This method is certainly a great advance over the older one and generally gave satisfactory results in our preliminary tests. It had, however, to be standardized particularly in reference to the concentration of the dicyanodiamide in solution and also to the acidity of the solution.

The nature of the decomposition of cyanamide in the soil.

Two series of flasks were prepared each containing 100 grammes samples of soil, the moisture content of which being the same, *viz.*, 15 per cent. In the first series 0·06 gramme of nitrolim equivalent to 10 milligrammes nitrogen was added to each flask.

The second series was sterilized by autoclaving the flasks for 30 minutes at a temperature of 125°C., and to each flask was then added 0·06 gramme of nitrolim previously sterilized in a small tube.

The two series were, therefore, comparable except that the first contained the ordinary soil organisms while the second was sterile. The flasks, plugged with cotton wool, were then kept at room temperature and analyses made at intervals to ascertain the form in which the nitrogen was present. In this experiment nitrites and nitrates were not estimated, the decomposition being followed to ammonia stage only.

Later experiments indicate that sterilizing the soil at a high temperature may modify the behaviour of the latter. A better comparison would therefore be to sterilize both series and re-inoculate one.

Table VIII.

Showing the degradation of cyanamide in sterilized and unsterilized soils.

(10 milligrammes cyanamide nitrogen per 100 grammes of soil.)

Nitrogen as	Sterilized soil			Unsterilized soil		
	40 hrs.	4 days	7 days	40 hrs.	4 days	7 days
	per cent.	per cent.	per cent.	per cent.	per cent.	per cent.
Cyanamide ..	23·0	4·0	0·00	1·1	0·00	0·00
Dicyanodiamide ..	0·0	0·0	0·00	0·0	0·00	0·00
Urea	Not determined		100·00	Not determined		3·73
Ammonia ..	Do.	Do.	0·00	Do.	Do.	6·17

In the case of sterilized soil no ammonia could be found even after 15 days.

In a second experiment the method of sterilizing the soil and manure, etc., was the same as before but the amount of cyanamide added was increased, being equivalent to 83 milligrammes N per 100 grammes of soil, a little over 8 times the amount used in the first experiment. The samples were analysed at the end of 14 days.

TABLE IX.

Showing milligrammes of nitrogen found in various forms after 14 days.

(83 milligrammes of cyanamide N per 100 grammes of soil.)

	Sterilized soil	Unsterilized soil
As Cyanamide ..	2·5	0·055
,, Dicyanodiamide ..	23·0	11·3[illegible]
,, Urea	45·0	6·300
,, Ammonia	0·0	34·700

These results indicate that the decomposition of cyanamide in the soil under investigation is of a purely chemical nature up to the urea stage and that the hydrolysis of urea and the subsequent oxidation of the resulting ammonia are of a biochemical nature, thus confirming the observations of Ulpiani[1] and Cowie. The fact that the velocity of degradation in the soil sterilized at 125°C. is slower than that in the unsterilized soil and that it is slower still when the soil is ignited suggests that the transformation is one of physico-chemical nature. It will be noticed that in the first experiment when the concentration of nitrolim was normal there was no evidence of the ormation of dicyanodiamide. In the second case when high concentrations of nitrolim were employed considerable quantities were produced.

[1] Ulpiani. *Gazetta Chemica Italica*, **40**, 1910, 613, 666.

THE NATURE AND RATE OF FORMATION OF THE DECOMPOSITION PRODUCTS.

A detailed study of the nature and quantities of the products formed and of the rate of decomposition was next made with garden and paddy soils which analysed as follows :—

	Garden soil F. No. 44	Paddy soil, Wetland Block A.
	Per cent.	Per cent.
Moisture	3·54	3·63
Loss on ignition	5·01	4·95
Insolubles	73·05	72·87
Iron oxide	3·93	5·19
Alumina	6·99	9·79
Lime	2·12	0·97
Magnesia	1·40	1·42
Total phosphoric acid ..	0·05	0·07
Total potash	0·62	0·48
Undetermined	3·29	0·63
TOTAL ..	100·00	100·00
Nitrogen	0·123	0·043
Available phosphoric acid ..	0·028	0·010
Available potash	0·024	0·011
Fine gravel	9·17	3·55
Coarse sand	27·26	20·09
Fine sand	13·93	27·77
Silt	10·63	11·52
Fine silt	23·63	26·04
Clay	11·22	7·26
Water Extract		
Lime	0·009	0·006
Magnesia	..	..
Sulphuric acid	0·019	0·010
Carbonic acid	0·020	0·018
Chlorine	0·029	0·007
Total solids	0·150	0·064

4,500 grammes of the garden soil were passed through a 2 mm. sieve of round holes and mixed with small quantities of water at a time so as to bring the moisture content to 20 per cent. of the soil. Into this 2·70 grammes of calcium cyanamide were thoroughly incorporated. The mixture contained 100 milligrammes of cyanamide nitrogen per kilogramme of soil. That the manure was properly incorporated into the soil was ascertained by taking a sample and determining its nitrogen content by the Kjeldahl-Gunning method. The mixture was put into a series of small pots and analysed at intervals.

In the case of the paddy soil small quantities of the mixture of soil and manure were put into small beakers. Water was added until the soil was submerged and the contents were then puddled.

The results obtained are given in Tables X and XI and plotted in Chart 1.

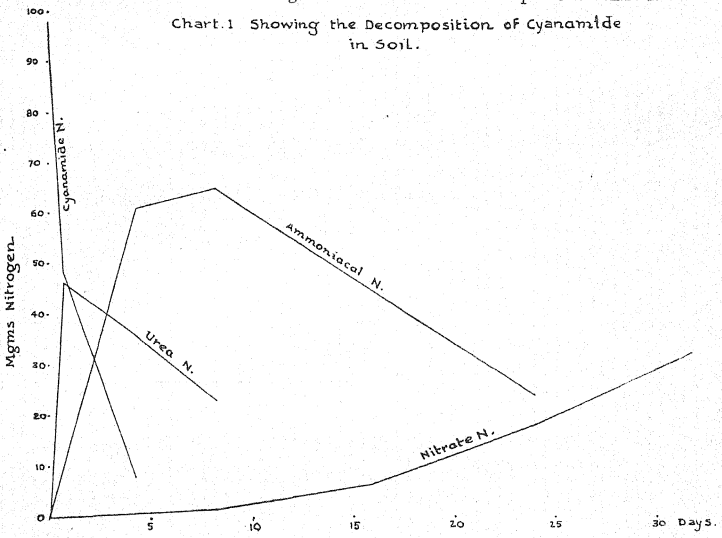

TABLE X.

Showing the decomposition of calcium cyanamide in the soil.

Garden soil F. No. 44.

(Cyanamide nitrogen 100 mg. per kilo of soil.)

	Initial	After 1 day	After 4 days	After 8 days	After 18 days	After 24 days	After 32 days
Cyanamide N ..	98·0	48·340	8·00	..	..	..	..
Dicyanodiamide N ..	..	..	..	..	..	..	..
Urea N	..	46·200	35·69	23·000	..	..	..
Ammonia N ..	..	..	60·82	65·000	..	24·30	..
Nitrite N	..	0·055	0·06	0·062	..	..	..
Nitrate N	..	..	..	1·300	6·37	18·22	32·0

TABLE XI.

Showing the decomposition of calcium cyanamide under puddled conditions.

(100 milligrammes of cyanamide N per kilo of soil.)

	After 2 days	After 25 days
Cyanamide N ..	6·30	....
Dicyanodiamide N ..	....	....
Urea N	16·80	8·40
Ammonia N	21·00	27·30

One striking feature of the results is that no dicyanodiamide can be detected when the quantity of manure applied is in consonance with agricultural practice. The results generally agree with those of Cowie. The decomposition of cyanamide in the soil is very rapid up to the urea and ammonia stages, the oxidation of ammonia to nitrates proceeding more slowly. Since nitrification proceeds at this slow rate there is little danger of all the nitrogen becoming available too quickly before it can be utilized by the crop. In the case of paddy soils, however, the conversion of the cyanamide nitrogen into ammoniacal nitrogen is very rapid, and we are not yet in a position to say how this affects the assimilation of the ammoniacal nitrogen by the paddy plant. Ammonia being fixed by the soil, there is little likelihood of loss taking place in this case, whereas if nitrates in garden soil were produced at an equal rate, loss would undoubtedly occur.

DICYANODIAMIDE AND ITS INFLUENCE ON PLANT GROWTH.

There has been a great deal of discussion in chemical literature concerning dicyanodiamide, and the views expressed by various investigators are of a conflicting nature both in regard to its formation in the soil and to its properties. Much of the data available is not entitled to serious consideration from an agricultural point of view owing to the fact that excessive quantities of nitrogen were used which would never be employed in practice. Moreover,

the method of analysis employed in some of the previous experiments are open to serious objection in that the precipitants used were not specific for dicyanodiamide. The problem is under thorough investigation by us and the preliminary results so far obtained may be discussed here.

In dosages ordinarily used in agricultural practice we have not been able to detect the presence of dicyanodiamide at any stage of the decomposition of calcium cyanamide. But when the dosage is considerably increased, dicyanodiamide is formed, the further decomposition of which seems to be very slow. For instance, when cyanamide nitrogen was applied to the soil at the rate of 83 milligrammes and 166 milligrammes per 100 grammes of soil, 11·5 milligrammes and 60 milligrammes respectively were found after 14 days and 4½ months respectively.

Form and rate of N applied per 100 grammes soil	Analysis carried out after	Milligrammes of dicyanodiamide found
	Days	
10 milligrammes cyanamide N	1	0·0 or 0 per cent.
83 Do. do.	14	11·5 or 25 ,,
166 Do. do.	135	60·0 or 36 ,,
13 milligrammes of pure dicyanodiamide N ..	2	10·0 or 77 ,,
20 Do. do.	135	1·0 or 5 ,,

While it is admitted that the evidence before us is insufficient to make a definite statement in regard to the formation of dicyanodiamide during the decomposition of cyanamide in the soil, we may form some conclusions having regard to the conditions that are known to favour its production.

We see from the statement above that dicyanodiamide, whether formed from cyanamide or added in the pure state, persists as such in the soil for some time, and as we have not been able to detect it at any stage of the decomposition when cyanamide is added in ordinary agricultural doses, it is probable that

under such conditions no dicyanodiamide is formed. The fact that dicyanodiamide is formed when high concentrations of nitrolim are employed shows that there is a limit of safety in the use of this fertilizer.

Moreover, it indicates the necessity for thorough incorporation of the manure in the soil when applied, otherwise local areas of high concentration may easily be formed leading to the production of dicyanodiamide. This, perhaps, explains why manuring with cyanamide even in ordinary dosages is sometimes reported to be harmful.

We may also form some idea as to why under these conditions dicyanodiamide should form in the soil. Cyanamide in an acid or alkaline medium polymerises into dicyanodiamide. When calcium cyanamide is added to the soil, in contact with the soil moisture it resolves into cyanamide and calcium hydroxide. If the dosage be small and the manure thoroughly incorporated into the soil, no dicyanodiamide is formed. If, on the other hand, the dosage is large or the manure is not thoroughly and uniformly mixed with the soil, a relatively high percentage of calcium hydroxide will be formed in those areas where the manure happens to be concentrated. It is quite likely that this local concentration of alkali may be sufficient to bring about polymerisation of the cyanamide into dicyanodiamide. The hydrolysis of the calcium cyanamide and the amount of free calcium hydroxide present in the soil seem to be the factors determining the formation of dicyanodiamide. This view is based only on theoretical considerations and needs, of course, to be confirmed by further observations. As regards the fate of dicyanodiamide formed in, or added to, the soil, we have seen that it does not remain as such permanently but slowly changes into some other compounds, the chemical nature of which we are not yet in a position to explain. Experiments to induce the rapid disappearance of dicyanodiamide from the soil show signs of promise. More than this cannot at present be said.

Toxic influence of dicyanodiamide.

Dicyanodiamide even in such large doses as 5 cwt. per acre has not affected germination in our trials with sixteen varieties of cereals and pulses commonly cultivated in South India. A few days after germination, however, all the plants showed signs of nitrogen starvation. When nitrogen, however, was applied in the form of potassium nitrate, the plants began to grow again normally. Our results agree in this respect entirely with those of Cowie in regard to the influence of dicyanodiamide on germination and growth.

TABLE XII.

Showing the effect of dicyanodiamide on germination and growth of plants.

(The salt was applied in the form of a solution of pure dicyanodiamide to small quantities of soil in pots at rates representing 1 cwt., 3 cwt. and 5 cwt. per acre.)

S. No.	Local or popular name	Botanical name	Control	DICYANODIAMIDE	
				Germination	Growth 15 days after sowing
				1, 3 & 5 cwt.	1, 3 & 5 cwt.
1	Wheat ..	*Triticum* sp. ..	All germinated well and growing well after a fortnight.	All germinated and growing well.	All showing signs of nitrogen starvation. *Ragi* seems to be most affected. The addition of potassium nitrate to the soil revived the plants and growth was again normal.
2	Gingelly ..	*Sesamum indicum* ..			
3	Paddy ..	*Oryza sativa* ..			
4	Cholam ..	*Sorghum vulgare* ..			
5	Red-gram ..	*Cajanus indicus* ..			
6	Horse-gram ..	*Dolichos biflorus* ..			
7	Ragi ..	*Eleusine coracana* ..			
8	Bengal-gram	*Cicer arietinum* ..			
9	Black-gram ..	*Phaseolus mungo* ..			
10	Samai ..	*Panicum miliare* ..			
11	Cow pea ..	*Vigna catjang* ..			
12	Cumbu ..	*Pennisetum typhoideum*			
13	Tenai ..	*Setaria italica* ..			
14	Kudurai vali	*Panicum crus-galli* ..			
15	Panivaragu ..	*Panicum miliaceum* ..			
16	Lab lab ..	*Dolichos lablab* ..			

A soil, to which dicyanodiamide was added at the rate of 5 cwt. per acre, grew ragi (*Eleusine coracana*) splendidly after six months. The photographic reproductions are given in Plate I, fig. 4. The dicyanodiamide in this period has therefore been converted into harmless products.

We have just now seen that the adverse influence of dicyanodiamide on the crop is to prevent the supply of nitrogen to it. This means that the process of

nitrification is inhibited or retarded. The results of experiments to check this are embodied in Table XIII and graphically represented in Chart II.

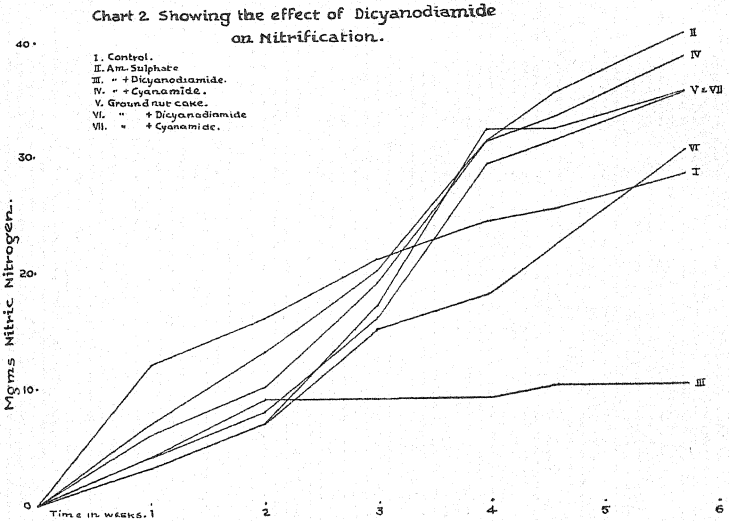

TABLE XIII.

Showing the influence of dicyanodiamide on nitrification.

(50 milligrammes of nitrifiable N per kilo of soil.
25 milligrammes of dicyanodiamide N per kilo of soil.)

Nature of treatment	Milligramme of nitrate N found after days					
	7	14	21	28	32	40
Control	12·0	16·0	21·0	24·0	25·0	28·0
Ammonium sulphate	7·0	13·0	20·0	31·0	35·0	40·0
Ammonium sulphate and dicyanodiamide	4·0	9·0	9·0	8·8	10·0	9·8
Ammonium sulphate and cyanamide	6·0	10·0	19·0	31·0	33·0	38·0
Groundnut cake	3·0	7·0	17·0	32·0	32·0	35·0
Groundnut cake and dicyanodiamide.	2·8	6·9	15·0	18·0	22·0	30·0
Groundnut cake and cyanamide ..	4·0	8·0	15·7	29·0	31·0	35·0

It will be seen that our results again generally confirm Cowie's observations. In our experiments the rate of nitrification, where this occurred, was, as would be expected, more rapid than in Cowie's experiments. One very interesting fact to be noted is that dicyanodiamide does not seem to affect the nitrification of groundnut cake so drastically as that of ammonium sulphate. Cowie also had a similar experience in the nitrification of dried blood.

There would appear to be some evidence that in the presence of decomposing organic matter the dicyanodiamide either undergoes change itself or at any rate its toxicity towards the nitrifying organisms is very largely reduced.

Support for this hypothesis is found in the observation we have made that dicyanodiamide when added to cattle dung rapidly disappears though there is practically no change if the urine alone is used.

If confirmed, this observation may lead to results of considerable practical importance in the use of nitrolim as a fertilizer.

Summary.

1. Calcium cyanamide when applied to the soil in ordinary agricultural doses is not harmful to crops.

2. No lapse of time between the application of the manure and sowing is necessary.

3. Calcium cyanamide rapidly decomposes in the soil into urea, this change being chemical or physico-chemical in nature; the hydrolysis of urea to ammonia and its subsequent oxidation to nitrates is a biochemical process.

4. Dicyanodiamide is not produced in the soil from calcium cyanamide when it is applied in normal doses and thoroughly incorporated in the soil, but may be produced when high concentrations of nitrolim are employed.

5. Dicyanodiamide decomposes in the soil with considerable slowness.

6. Dicyanodiamide does not affect germination, but inhibits nitrification. This inhibition is more pronounced in the case of ammonium sulphate than in the case of organic nitrogenous substances such as groundnut cake.

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MEMOIRS OF THE DEPARTMENT OF AGRICULTURE IN INDIA

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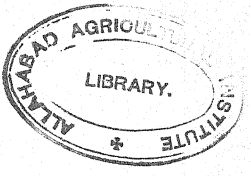
AND

PANDIT LAL CHAND DHARMANI, L.Ag
Assistant to Agricultural Chemist to Government, Punjab



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[Received for publication on 2nd November, 1923.]

Introductory.

The experiments recorded in the following paper are the result of an endeavour to obtain information on the feeding values of some of the commoner Indian foodstuffs as shown by actual digestibility trials.

Although a large number of analyses of feeding stuffs have been made in this country, notably by Leather,[1] yet little or nothing has yet been done to study systematically and side by side the values of feeding stuffs as shown by chemical analysis and their values as indicated by digestion trials on animals.

Chemical analysis by itself, while giving an indication of the actual amounts of various food ingredients present, such as the fats, proteins, carbohydrates, etc., can tell us but little of the way in which any particular animal will be able to utilize them for its general metabolism; and a foodstuff, the chemical analysis of which shows a high content of fats, carbohydrates and proteins, may be of less value to an animal than one with a "lower chemical value" if the contents of the latter are more digestible than those of the former.

In times of scarcity it is a matter of great necessity to find materials not ordinarily used, which may be utilized to supplement an animal's ration.

[1] Leather, J. W. *Agri. Ledger*, 10 (1901) and 7 (1903).

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Chemical analysis by itself, while giving an indication of the actual amounts of various food ingredients present, such as the fats, proteins, carbohydrates, etc., can tell us but little of the way in which any particular animal will be able to utilize them for its general metabolism; and a foodstuff, the chemical analysis of which shows a high content of fats, carbohydrates and proteins, may be of less value to an animal than one with a "lower chemical value" if the contents of the latter are more digestible than those of the former.

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Such a case presented itself in the Punjab when the first author was asked in 1921 to report on the value of *shisham* (*Dalbergia sissoo*) leaves as a supplementary article of diet for animals.

The analysis of fresh *shisham* leaves collected in July of that year showed them to possess a feeding value comparable to that of oats, but when feeding trials were undertaken, an entirely different picture was presented, animals not being able to tolerate more than five to six lb. daily with impunity, as will be shown later.

The problem before us is to determine what percentage of the various ingredients which chemical analysis reveals, can be digested and utilized by the animal, and presents different aspects according to whether we are dealing with growing or mature animals.

In the case of mature animals the digested food material has only to be utilized for external and internal work and for the general wear and tear of the organism, whereas young animals have in addition to lay on extra tissue material, and in this latter connection it is the *type* or quality of food material which is most important, quite apart from its quantity and digestibility. Whereas the fats and carbohydrates are of a comparatively simple chemical composition and few in number, and may within certain limits be considered of equal value for the nutrition of growing or mature animals, the protein part of the food is much more complicated. With growing animals the most important point is, not the actual quantity of protein which the food contains, but its quality, or, in other words, the nature of the amino acids into which the protein is broken down in the course of digestion.

If a growing animal is fed a standard ration in which all of the protein is fed in the form of Indian corn or maize, growth rapidly comes to a standstill and ultimately death ensues, or again if a pregnant cow is fed such a diet, the calf will be born prematurely, be imperfectly formed and but short-lived. In both cases deficiency in the diet is due to the fact that certain amino acids, *viz.*, tryptophane and glycine, which are absolutely necessary for the formation of new growing tissues, are absent. Thus studies in animal foodstuffs should be conducted along three main lines. First, the crude chemical analysis carried out in the laboratory to give us a quantitative picture; secondly, digestion trials which should indicate how far the animal can digest these same ingredients; and, thirdly, what may be termed the biological investigation of the proteins present, and their value as determined by experiments on young growing animals. All these are highly important both from a nutritive and from an economic standpoint, the problem presenting different aspects according to whether we are dealing with young or mature animals. A glance

at the work which has already been done in Europe and America will indicate the magnitude of the problems to be tackled, but it does not follow that results obtained from any of the above mentioned lines of enquiry obtained in one country will hold good in another in which climatic and other conditions vary widely. Hence it is of the utmost importance for Indian agriculture that systematic research along the lines indicated should be attempted.

It is felt that some apology is perhaps needed for presenting some of the results obtained, results which though negative and impossible in themselves nevertheless give us information of a very positive kind. Such results will be indicated and discussed in the context; for example, negative digestibility coefficients and coefficients greater than one hundred.

No mention will be made here of the extensive subject of vitamines as the investigation was confined to chemical analysis and digestibility coefficients.

It was a matter of great regret that only two animals were available for the work in 1921-22, both of which were rather old and feeble, and the extraordinary results obtained on the *bhusa* diet alone, which must also have affected the succeeding diets, led to a repetition of the *bhusa* trials during the winter of 1922-23 on better conditioned animals, but unfortunately again only two were available.

Too much emphasis cannot be laid on the fact that work of this description must be carried on with as large a number of animals as possible, though this entails a vast amount of work in supervision and analysis.

Object and method of the experiments.

The object in view has been to determine the quantitative feeding value and the digestibility coefficients of *bhusa*, gram, maize, and fresh *shisham* leaves. The *bhusa* and *shisham* leaves may be considered as roughages, while gram and maize are concentrates. An attempt was made to determine the digestibility coefficient of *bhusa* alone, and better results would perhaps have been obtained had the animals been younger and in better condition. The method employed with such a roughage as *bhusa* was to analyse both the *bhusa* given and the dung voided during the experimental period and calculate the digestibility coefficients directly from the results.

This is very simple but the digestibility of concentrates by herbivora is more complicated since they cannot be fed as the sole feed of these animals. They must therefore be fed along with a known amount of a roughage whose digestibility by the same animal has been determined in a preceding period. From the digestibility of the total ration and the known digestibility of the

roughage that of the concentrate is obtained by means of a second calculation by difference.

The sequence of diets employed was :—*Bhusa*, *bhusa* and gram, *bhusa* and maize, and *bhusa* and *shisham* leaves. While the *shisham* leaves may be considered as a roughage it was impossible to feed animals on the fresh leaves alone, not more than five to six pounds being acceptable even when fed with *bhusa*. The animals were fed for a period of 10 days on each diet under investigation before the actual chemical and other data were determined (in order to remove the effects of previous feeding).

The feeding of the first preparatory period was commenced on the 29th November, 1921, and carried on till the 7th December for both animals, Sawa and Gora by name. During the period of 9 days *bhusa* alone was given, and the amount of *bhusa* eaten, the dung voided, and the daily weights of the animals were carefully recorded.

The actual experimental period was from the 7th December till the 23rd during which time the same records were made and detailed analyses were made of the daily *bhusa* given and the dung voided.

We were thus able to determine the total quantities of each food constituent, such as fats, proteins, etc., present both in the food eaten and in the dung voided, although, as will be pointed out later, some very abnormal results were obtained.

Method of feeding the animals. The animals were kept in a stable and the food placed in zinc troughs so constructed that none was lost, and each animal was confined to its own trough. About 16 lb. of *bhusa* was finely sieved in order to separate dust, and this was placed in the troughs in three separate parts, in the morning, at noon and in the evening, and on the following morning any residue left was collected and carefully weighed. The animals were watered twice daily, once after the daily weighing which took place before they were fed, and once after the evening meal. Common salt was always available. The dung was collected in bags fixed on to the animal so arranged that no contamination with urine took place—as shown in Fig. 1—both morning and evening, and the daily yield carefully sampled and analysed. During the course of the experiments the animals were stall-fed and did no work other than walking up to the weigh-bridge 200 yards away and back.

Period of feeding on bhusa. During this period of 17 days Sawa ate 166 lb. of *bhusa* corresponding to 156·5 lb. of dry matter, the analysis of which is shown in Tables I and Ia, the amount of dung collected being 381 lb. corresponding to 83 lb. of dry matter. The analyses of dung are also shown. The corresponding figures for Gora were :—*Bhusa* eaten 148 lb. corresponding to

139·6 lb. of dry matter and the total quantity of dung voided was 308·5 lb. corresponding to 77·12 lb. of dry matter. From the total dry matter in the

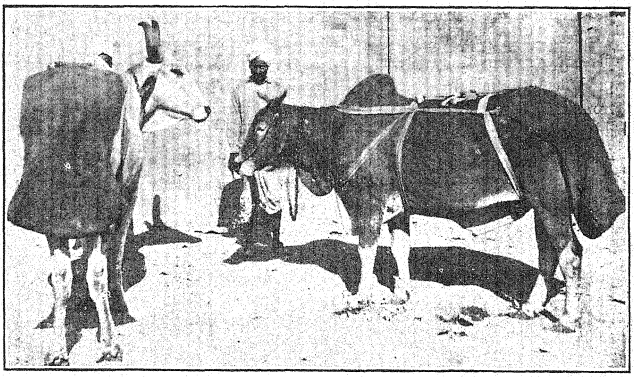


FIG. 1. Bullocks Sawa and Gora with harness for collecting dung.

bhusa and that in the dung voided the digestibility coefficient of the *bhusa* found is 46·93 for Sawa, and 44·76 for Gora.

The digestibility coefficients of the various ingredients of the *bhusa* were also determined from the analyses of *bhusa* and dung, and some rather extraordinary results were obtained. From the tables we notice that the ash, fat and protein which are present in the original *bhusa* all show negative digestibility coefficients, indicating that they are present in greater quantities in the fæces than in the food given. This is intelligible, when dealing with *bhusa* such as that used which contains but a small percentage of those substances, when it is remembered that certain excretory products from the animals are also voided in the fæces which have been ignored in the experiment but which are obviously included in the indigestible fraction of the food.

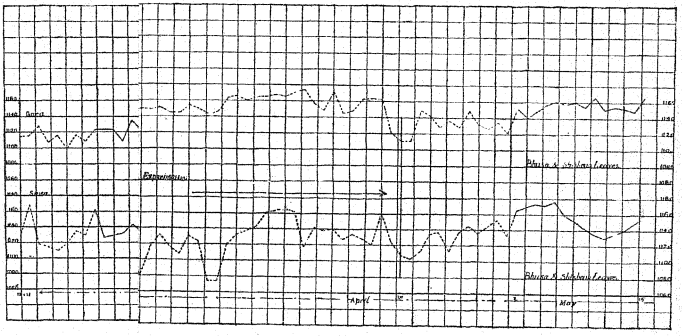
The crude fibre and nitrogen-free extract which are present in greatest proportions in the *bhusa* show no such anomalies, as neither of these would be derived from the excretory products of the animal. Such negative results are not uncommon, when investigating substances present in only small quantity, and, as Armsby[1] has shown, when investigating the coefficients for concen-

[1] Armsby. Errors of digestibility. *Amer. Jour. Sci.*, **29**, 355, 1885.

trates mixed with roughage the range of uncertainty introduced may be very great and such negative results may occur.

Both animals show these abnormalities to a similar degree. An interpretation of these figures indicates that the crude fibre and carbohydrates are between 50 and 60 per cent. digestible, the digestibility of the very small percentages of fats and proteins being overmasked by the presence of excretory products contaminating the fæces. In view of the negative figures obtained from these two animals it was decided to repeat this trial, using better animals, and the test was accordingly carried out in January and February of the following year, the results of which will be recorded in full detail later.

Bhusa and gram trials. From the 24th December until the 15th January, the transition period, the animals were fed on 4 lb. of ground gram per day in addition to *bhusa*. The gram, in a coarsely crushed condition, was fed to the animals in the morning, mixed with some *bhusa*, so that practically the whole of the *bhusa* and the gram was eaten. Additional *bhusa* up to a total of 16 lb. was then fed to them during the rest of the day. From the 16th to the 29th, the experimental period, the animals were fed 4 lb. of gram daily, all of which was eaten, and 16 lb. of *bhusa*. During these 14 days each animal ate 56 lb. of gram. Sawa ate in addition 182 lb. of *bhusa* corresponding to 171·6 lb. of dry matter, whilst Gora ate 164·5 lb. of *bhusa* corresponding to 155·1 lb. of dry matter. Sawa excreted 471·5 lb. of dung corresponding to 90·34 lb. of dry matter, while Gora excreted 412·5 lb. of dung corresponding to 94·75 lb. of dry matter. From these two series of data the digestibility coefficient of the combined feed in the case of Sawa was found to be 59·46, whilst in the case of Gora it was 54·07 on the dry matter of the combined feed. In Tables II and IIa are shown the analyses of the *bhusa*, gram and dung and the digestibility coefficients of the various ingredients of the combined feed. In view of the negative values given for the *bhusa* figures it seems perhaps invidious to work out the coefficients of the gram alone. If, however, we assume that the digestibility of the *bhusa* was unaltered by the addition of the gram, an assumption which is not strictly justified, it is possible to compute how much of each kind of digestible matter, protein, crude fibre, nitrogen-free extract, etc., in the total ration, was derived from the *bhusa*; the remainder must therefore have come from the gram. The percentage of digestibility can thus be determined. These figures are shown in Tables II and IIa. Turning to these tables we notice that some of the figures are very high, being above 100 per cent., the interpretation of which is that the influence of the gram on the *bhusa* is to render some of the ingredients of the latter still more digestible in the combined diet than alone, this added digestibility thus being added on to that of the gram. In the


Expiration
April
May

case of Sawa, the gram as a whole (to a small extent) and the ash and the fibre (to a greater extent, as they are present in small amount) all show this effect and to a certain degree also the nitrogen-free extract, whereas in the case of Gora these figures are not so high and approximate more nearly to what one would expect. It may be mentioned that during the course of the experiment Gora was in better physical condition than Sawa besides being somewhat younger.

The bhusa and maize trial. From January 29th to February 9th was a transition period during which 4 lb. of maize and 16 lb. of *bhusa* were fed to the animals daily. The maize was fed by grinding it up, moistening it and mixing it with half the daily ration of *bhusa* and fed at 8 A.M., the remaining *bhusa* being fed at 6 P.M. in the evening. On February 9th the experimental period commenced and continued until February 22nd, the same feeding régime being observed. It was found that by mid-day the whole of the maize given in the morning meal with the mixed *bhusa* was entirely eaten, any residue being negligible. During this period each animal was fed 56 lb. of maize corresponding to a total of 50·97 lb. of dry matter. Sawa ate in addition 122 lb. of *bhusa* corresponding to 115 lb. of dry matter and Gora ate in addition 121 lb. of *bhusa* corresponding to 114 lb. of dry matter. During this period Sawa voided 309 lb. of dung corresponding to 60·7 lb. of dry matter and Gora 280·5 lb. corresponding to 70·5 lb. of dry matter. The digestibility coefficient of the combined feed in the case of Sawa was 63·4 and the digestibility coefficient in the case of Gora was 57·28, a decided increase over the last two coefficients obtained. The results are shown in Tables III and IIIA. Looking at the digestibility coefficients of the individual constituents of the combined diet we notice that only the ash (in the case of Sawa) presented an abnormal figure (being negative), whilst those of Gora all show a positive value. When, however, we come to compute the digestibility coefficients of the maize as a whole and of its various constituents, we again find some very high figures. The maize as such shows 100 per cent. digestibility whilst the figures for the ash and crude fibre are considerably higher as they are also in the case of gram. This can only mean that the continued effect of feeding the animals on a partly cereal diet is having a considerable influence on the digestibility of the *bhusa* fed.

At the end of this period on the 22nd February the animals were returned to the farm until the 20th April, during which time a record of their weights was kept as shown in Fig. 2. They were fed on ordinary farm rations—wheat *bhusa, senji* (*Melilotus parviflora*), cane tops (*Saccharum officinarum*) and gram (*Cicer arietinum*)—and did very little work during the whole of this period.

The bhusa and shisham period. The period from the 20th April to the 1st May was preparatory during which time the animals were fed as much *shisham* leaves for the first few days as they cared to eat; after which the amount they were allowed to eat was restricted to about 5 to 6 lb. a day. They were also given in addition from 8 to 10 lb. of *bhusa* daily. During this preparatory period it was found that the animals ate the leaves with great avidity to begin with, but their appetite for large quantities soon diminished and they began to pass watery voidings. On account of this, as mentioned above, the daily ration of leaves was reduced to 6 lb. and one oz. of ammonium carbonate per day was administered for 4 days, as a result of which treatment the animals were rapidly cured. During the preparatory period the daily body weights were kept and fodder residues were carefully noted, but no dung was collected. The actual experimental period was from the 2nd to the 15th May when fresh *shisham* leaves were mixed with *bhusa* and fed to the animals in the morning; more *bhusa* being again fed to them in the evening but without leaves. No food was given during the night. The animals were watered 3 times a day, first after weighing, at noon, and between 4 and 5 P.M. During this period the animals ate the whole of the *shisham* leaves that were given; Sawa ate 64 lb. of *shisham* corresponding to 18·21 lb. of dry matter, and in addition 121·5 lb. of *bhusa* corresponding to 114·6 lb. of dry matter. During the same time Gora ate 68 lb. of *shisham* corresponding to 19·35 lb. of dry matter and 137·5 lb. *bhusa* corresponding to 130 lb. of dry matter. The complete data are shown in Tables IV and IVA. Sawa during this period voided 327 lb. of dung corresponding to 54 lb. of dry matter and Gora 308 lb. corresponding to 63 lb. The digestibility coefficient of the combined feed in the case of Sawa was 59·2 and in the case of Gora 57·6. Turning now to the tables we notice that the figures for the individual constituents of the combined diet are all perfectly normal and agree fairly closely for the two animals, there being no very wide discrepancies between them. When, however, we come to calculate the co-efficients for the individual constituents of the *shisham* leaves and the *shisham* as a whole, we again notice very abnormal figures. The coefficients for the *shisham* work out at 136 per cent. in one case and 143 per cent. in the other, whereas in both cases the figures for ash, fats, crude fibre, proteins and nitrogen-free extract are very high, and can in no wise be taken as absolute. These are very interesting. Had the figures for the *shisham* shown a negative value we would have interpreted it as meaning that the leaves could not be utilized by the animals, but being so highly positive, although the figures offer no actual criterion as to the true digestibility coefficient for the *shisham*, they yet

indicate that the very low negative values obtained when *bhusa* is fed alone are being ameliorated and converted into positive products, or, in other words, that an admixture of *shisham* with *bhusa* considerably augments not only the digestibility of the combined feed but also renders the *bhusa* itself more digestible.

Some investigations on feeding silaged *shisham* leaves have been carried out by Messrs. Branford and Sewell,[1] at the Government Cattle Farm, Hissar, and the conclusions which they come to are that animals readily eat *shisham* leaves which have been silaged, and that this material forms a very valuable adjunct to *bhusa* in times of fodder scarcity. These gentlemen have not carried out detailed observations in regard to the weights of the animals day by day nor were they able to carry out chemical analyses. Their results are in harmony with the practical experience of zemindars who find that animals will readily eat and tolerate small quantities of fresh *shisham* leaves, whereas larger amounts produce digestive disturbances. The effect is not so much poisonous but rather due to the fact that the young leaves contain some bitter and astringent principles which have a deleterious effect on the digestive tract, but this effect may be remedied by keeping the quantity of fresh leaves eaten down to about 6 lb. per diem, and would appear to diminish as the leaves get older. The experiments carried out at Hissar with siloed leaves seem to indicate that if the leaves are properly siloed the effect of the fermentative and other enzymic actions is to destroy the bitter principles mentioned above, and that siloed leaves would not only be less injurious to animals but would also be able to be fed in somewhat greater bulk to supplement ordinary rations. Experiments are in course of progress to investigate the alterations in the chemical ingredients of *shisham* leaves during the siloing period and it is hoped that these will be followed up by feeding and digestion trials.

The animals' weights during the course of the trials.

Each animal was weighed daily throughout the experimental and intermediate non-experimental periods, and the weights are plotted in Figs. 2 and 3, with explanatory notes. From November 29th, 1921, to December 23rd, when the animals were on a *bhusa* diet alone it will be noted that the initial and final weights in the case of Sawa were 1,130 lb. and 1,126 lb., whereas the highest and lowest weights recorded were 1,168 and 1,097 lb., the corresponding figures for Gora being 1,114 and 1,104 lb., and 1,134 lb. and 1,089 lb.

[1] Branford, R., and Sewell, E. Feeding Experiments at Govt. Cattle Farm, Hissar. *Pusa Agri. Res. Inst. Bull.* 130.

year's trials. This is undoubtedly due to the fact that the animals were in far better condition and also that the *bhusa* was much richer in nutritive contents. It is interesting to compare the figures obtained in these *bhusa* trials with the figures of a similar experiment conducted with hay as given by Armsby. Table VII shows such a comparison of figures from one of Armsby's experiments with the figures obtained from the animal Mina in this second set of *bhusa* trials. There is no doubt whatever that the age and

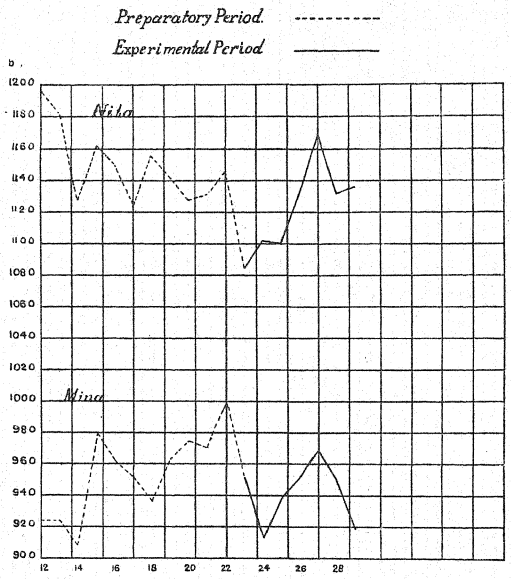


FIG. 3.

condition of the animals under trial very materially affect the results which will be obtained, and that the higher the percentage of the various nutritive ingredients present in a roughage such as *bhusa*, the less likelihood is there of obtaining absurd results such as were obtained in the first trials with *bhusa*.

These results though incomplete in themselves are presented as it is felt that they are of decided interest as indicating the initial lines along which work on feeding stuffs in India should proceed.

They will be continued and extended as opportunity offers.

Digestibility Experiments.

Table I.

Feed		*Bhusa* (from wheat) alone.
Animal		Sawa.
Period		7th to 23rd December, 1921.
Bhusa given ..	..	266 lb.
Bhusa residue ..	..	100 ,,
Bhusa eaten ..	..	166 ,, = 156·50 lb. dry matter.
Dung ..	..	381 ,, = 83·06 ,, ,, ,,
Digestibility coefficient	..	= 46·93.

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Dung	78·20	21·80	4·50	0·32	6·68	1·07	9·23

Actual quantities of different constituents.

In *bhusa* given ..	..	156·50	12·58	1·20	66·18	2·86	73·70
In dung voided ..	..	83·06	17·14	1·22	25·45	4·08	35·17
Bhusa digested (by difference) ..	..	73·44	−4·56	−0·02	40·73	−1·22	38·53
Digestibility coefficients	..	46·93	−36·25	−1·67	61·53	−42·65	52·26

Digestibility Experiments.

Table Ia.

Feed	.. ..	*Bhusa* (from wheat) alone.
Animal	.. ..	Gora.
Period	.. ..	7th to 23rd December, 1921.
Bhusa given	.. ..	262 lb.
Bhusa residue	.. ..	114 ,,
Bhusa eaten	.. ..	148 ,, = 139·60 lb. dry matter.
Dung	.. ..	308·5 ,, = 77·12 ,, ,, ,,
Digestibility coefficient	..	= 44·76.

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Dung	75·00	25·00	5·01	0·36	7·81	1·20	10·62

Actual quantities of different constituents.

In *bhusa* given ..	..	139·60	11·22	1·07	59·00	2·55	65·71
In dung voided ..	..	77·12	15·46	1·11	24·10	3·70	32·76
Bhusa digested (by difference) ..	..	62·48	–4·24	–0·04	34·90	–1·15	32·95
Digestibility coefficients	..	44·76	–37·79	–3·74	59·13	–45·09	50·13

Digestibility Experiments.

Table II.

Feed .. .. *Bhusa* and gram.
Animal .. .. Sawa.
Period .. .. 16th to 29th January, 1922.

Bhusa given	224 lb.			
Bhusa residue	42 „			
Bhusa eaten	182 „	= 171·60	lb.	dry matter.
Gram eaten	56 „	= 51·30	„	„ „
Total ..		222·90	„	„ „
Dung	471·5 lb.	= 90·34	„	„ „
Digestibility coefficient of combined diet		= 59·46		
„ „ „ gram		= 101·40		

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Gram	8·42	91·58	3·29	2·62	6·90	18·88	59·89
Dung	80·84	19·16	3·68	0·42	5·35	1·45	8·26

Actual quantities of different constituents.

In *bhusa* given	171·60	13·80	1·31	72·56	3·13	80·81
In gram given	51·30	1·84	1·47	3·86	10·57	33·54
In total feed given	222·90	15·64	2·78	76·42	13·70	114·35
In dung voided	90·34	17·36	1·98	25·23	6·84	38·95
Total feed digested (by difference)	132·56	−1·72	0·80	51·19	6·86	75·40
Total digested from *bhusa*	80·54	−5·00	−0·02	44·62	−1·34	42·24
Total digested from gram	52·02	3·28	0·82	6·57	8·20	33·16
Digestibility coefficients of constituents of combined feed ..	59·46	−11·00	28·78	66·98	50·07	65·92
Digestibility coefficients of constituents of gram	101·40	178·30	55·78	170·20	77·58	98·85

Digestibility Experiments.

Table IIA.

Feed .. .. *Bhusa* and gram.
Animal .. .. Gora.
Period .. .. 16th to 29th January, 1922.

Bhusa given	224 lb.		
Bhusa residue	59·5 „		
Bhusa eaten	164·5 „	= 155·10 lb.	dry matter.
Gram eaten	56·0 „	= 51·29 „	„ „
Total ..		206·39 „	„ „
Dung	412·5 „	= 94·75 „	„ „
Digestibility coefficient of combined diet		= 54·07	
„ „ „ gram		= 82·30	

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Gram	8·42	91·58	3·29	2·62	6·90	18·88	59·89
Dung	77·03	22·97	4·23	0·48	5·91	1·87	10·48

Actual quantities of different constituents.

In *bhusa* eaten	155·10	12·47	1·18	65·59	2·83	73·04
In gram eaten	51·29	1·84	1·47	3·86	10·57	33·54
In total feed eaten	206·39	14·31	2·65	69·45	13·40	106·58
In dung voided ..	94·75	17·45	1·93	24·38	7·71	43·23
Total digested (by difference)	111·64	−3·14	0·67	45·07	5·69	63·35
Total digested from *bhusa*	69·42	−4·71	−0·04	38·80	−1·28	36·61
Total digested from gram	42·22	1·57	0·71	6·27	6·97	26·74
Digestibility coefficients of constituents of combined feed ..	54·07	−21·94	25·28	64·89	42·46	59·44
Digestibility coefficients of constituents of gram	82·30	85·30	48·30	162·40	65·95	79·72

Digestibility Experiments.

Table III.

Feed	..	..	*Bhusa* and maize.	
Animal	..	..	Sawa.	
Period	..	..	9th to 22nd February, 1922.	
Bhusa given	..	.. 142 lb.		
Bhusa residue	..	.. 20 ,,		
Bhusa eaten	..	.. 122 ,,	= 115·03 lb.	dry matter.
Maize eaten	..	.. 56 ,,	= 50·97 ,,	,, ,,
	Total	..	166·00 ,,	,, ,,
Dung	..	.. 309 ,,	= 60·72 ,,	,, ,,
Digestibility coefficient of combined diet			= 63·40	
,, ,, ,, maize			= 100·65	

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Maize	8·99	91·01	1·95	3·51	1·84	9·31	74·40
Dung	80·35	19·65	3·42	0·45	5·30	1·62	8·86

Actual quantities of different constituents.

In *bhusa* eaten	115·03	9·25	0·88	48·64	2·10	54·17
In maize eaten	50·97	1·09	1·97	1·03	5·21	41·67
In total feed eaten	166·00	10·34	2·85	49·67	7·31	95·84
In dung voided	60·72	10·57	1·39	16·38	5·01	27·38
Total feed digested (by difference)	105·28	−0·23	1·46	33·29	2·30	68·46
Total digested from *bhusa*	53·98	−3·35	−0·01	29·93	−0·90	28·31
Total digested from maize	51·30	3·12	1·47	3·36	3·20	40·15
Digestibility coefficients of constituents of combined feed ..	63·40	−22·24	51·22	67·00	31·45	71·41
Digestibility coefficients of constituents of maize	100·65	286·20	74·60	326·20	61·40	96·34

Digestibility Experiments.

Table IIIa.

Feed .. .. *Bhusa* and maize.
Animal .. .. Gora.
Period .. .. 9th to 22nd February, 1922.

Bhusa given ..	.. 141 lb.			
Bhusa residue ..	.. 20 „			
Bhusa eaten ..	.. 121 „	= 114·09 lb.	dry matter.	
Maize eaten ..	.. 56 „	= 50·97 „	„	„
Total ..		165·06 „	„	„
Dung ..	.. 280·5 „	= 70·51 „	„	„
Digestibility coefficient of combined diet		= 57·28		
„ „ „ maize		= 85·29		

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Maize	8·99	91·01	1·95	3·51	1·84	9·31	74·40
Dung	74·86	25·14	3·39	0·45	6·49	1·84	12·97

Actual quantities of different constituents.

In *bhusa* eaten	114·09	9·17	0·87	48·25	2·08	53·73
In maize eaten	50·97	1·09	1·97	1·03	5·21	41·67
In total feed eaten	165·06	10·26	2·84	49·28	7·29	95·40
In dung voided	70·51	9·51	1·26	18·21	5·16	36·38
Total feed digested (by difference)	94·55	0·75	1·58	31·07	2·13	59·02
Total digested from *bhusa*	51·07	–3·47	–0·03	28·53	–0·94	26·94
Total digested from maize	43·48	4·22	1·61	2·54	3·07	32·08
Digestibility coefficients of constituents of combined feed ..	57·28	7·31	55·62	63·03	29·21	61·85
Digestibility coefficients of constituents of maize	85·29	387·10	81·70	246·60	58·91	76·98

DIGESTIBILITY EXPERIMENTS.

TABLE IV.

Feed .. .. *Bhusa* and *shisham* leaves.
Animal .. .. Sawa.
Period .. .. 2nd to 15th May, 1922.

Bhusa given ..	.. 142 lb.		
Bhusa residue ..	.. 20·5 ,,		
Bhusa eaten ..	.. 121·5 ,,	= 114·56 lb.	dry matter.
Shisham leaves eaten	.. 64 ,,	= 18·21 ,,	,, ,,
TOTAL ..		132·77 ,,	,, ,,
Dung ..	.. 327 ,,	= 54·18 ,,	,, ,,
Digestibility coefficient of combined diet		= 59·20	
,, ,, ,, *shisham* leaves		= 136·25	

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Shisham leaves ..	71·54	28·45	2·95	1·13	5·59	5·11	13·68
Dung .. .	83·43	16·57	2·43	0·23	5·70	1·06	7·15

Actual quantities of different constituents.

In *bhusa* eaten	114·56	9·21	0·87	48·44	2·[illegible]	53·05
In *shisham* leaves eaten. ..	18·21	1·89	0·72	3·58	3·2[illegible]	8·76
In total feed eaten	132·77	11·10	1·59	52·02	5·36	62·71
In dung voided	54·18	7·95	0·75	18·64	3·47	23·38
Total feed digested (by difference)	78·59	3·15	0·84	33·38	1·89	39·33
Total digested from *bhusa*	53·78	–3·34	–0·01	29·81	–0·89	28·20
Total digested from *shisham* leaves	24·81	6·49	0·85	3·57	2·78	11·13
Digestibility coefficients of constituents of combined feed ..	59·20	28·38	52·83	64·16	35·25	62·71
Digestibility coefficients of constituents of *shisham* leaves ..	136·25	343·40	118·10	99·71	85·01	127·00

Digestibility Experiments.

Table IVa.

Feed .. .. *Bhusa* and *shisham* leaves.
Animal .. .. Gora.
Period .. .. 2nd to 15th May, 1922.

Bhusa given ..	.. 150 lb.			
Bhusa residue ..	.. 12·5 „			
Bhusa eaten ..	.. 137·5 „	= 129·65 lb.	dry matter.	
Shisham leaves eaten	.. 68 „	= 19·35 „	„	„
Total ..		149·00 „	„	„
Dung ..	.. 308 „	= 63·20 „	„	„
Digestibility coefficient of combined diet		= 57·56		
„ „	„ *shisham* leaves	= 143·35		

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Shisham leaves ..	71·54	28·45	2·95	1·13	5·59	5·11	13·68
Dung	79·48	20·52	2·60	0·27	7·48	1·09	9·08

Actual quantities of different constituents.

In *bhusa* eaten ..	..	129·65	10·42	0·99	54·82	2·36	61·05
In *shisham* leaves eaten	..	19·35	2·01	0·77	3·80	3·48	9·30
In total feed eaten ..	..	149·00	12·43	1·76	58·62	5·84	70·35
In dung voided ..	..	63·20	8·01	0·83	23·04	3·36	27·97
Total feed digested (by difference) ..	..	85·80	4·42	0·93	35·58	2·48	42·38
Total digested from *bhusa*	..	58·06	−3·94	−0·04	32·42	−1·06	30·61
Total digested from *shisham* leaves ..	..	27·74	8·36	0·97	3·16	3·54	11·77
Digestibility coefficients of constituents of combined feed ..	..	57·59	35·56	52·85	60·68	42·45	60·23
Digestibility coefficients of constituents of *shisham* leaves ..	..	143·35	415·80	126·00	83·14	101·70	126·50

Digestibility Experiments.

Table V.

Feed .. .. *Bhusa* (from wheat) alone.

Animal .. .. Nila.

Period .. .. 23rd January to 4th February, 1923.

Bhusa eaten .. .. 129 lb. = 118 lb. dry matter.

Dung .. .. 306 „ = 52·17 „ „ „

Digestibility coefficient of *bhusa* = 55·78 On dry matter.

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	8·54	91·46	7·08	0·740	41·36	3·70	37·85
Dung	82·95	17·05	2·64	0·175	6·09	1·00	6·97

Actual quantities of different constituents.

In *bhusa* given	117·98	9·13	0·955	53·36	4·77	48·83
In dung voided	52·17	8·08	0·536	18·64	3·06	21·33
Bhusa digested (by difference)	65·81	1·05	0·419	34·72	1·71	27·50
Digestibility coefficients	55·78	11·50	43·87	65·65	35·84	56·31

Digestibility Experiments.

Table Va.

Feed .. .. *Bhusa* (from wheat) alone.

Animal .. .. Mina.

Period .. .. 23rd January to 4th February, 1923.

Bhusa eaten .. .. 127·5 lb. = 116·60 lb. dry matter.

Dung .. .. 243 „ = 45·36 „ „ „

Digestibility coefficient of dry matter 61·10

Analytical composition.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa	8·54	91·46	7·08	0·740	41·36	3·70	37·85
Dung	81·33	18·67	3·07	0·185	6·02	1·12	8·04

Actual quantities of different constituents.

In *bhusa* given ..	..	116·60	9·03	0·944	52·73	4·72	48·26
In dung voided ..	..	45·36	7·46	0·450	14·62	2·72	19·54
Bhusa digested (by difference) ..	..	71·24	1·57	0·494	38·11	2·00	28·72
Digestibility coefficients	..	61·10	17·38	52·32	72·26	42·36	59·51

Digestibility Experiments.

Table VI.

(*a*) *Analysis of bhusa (from wheat), 1922 and 1923.*

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Bhusa 1922 ..	5·71	94·29	7·58	0·72	39·87	1·72	44·40
Bhusa 1923 ..	8·54	91·46	7·08	0·74	41·36	3·70	37·85

(*b*) *Albuminoid ratio and starch equivalents of different feeds used.*

...	Albuminoid ratio	Starch equivalent or food units
Wheat *bhusa*, 1922 .	1 : 43·04	50·50
Wheat *bhusa*, 1923 .	1 : 10·68	48·95
Gram .	1 : 3·49	113·64
Maize	1 : 8·86	106·43
Shisham leaves .	1 : 3·18	29·27

Digestibility Experiments.

Table VII.

The nutrition of Farm Animals, by Henry Prentiss Armsby, Ph.D., LL.D., (*page* 115), 1917.

	Moisture %	Dry matter %	Ash %	Ether extract %	Crude fibre %	Protein %	Nitrogen-free extract %
Analysis.							
Hay	15·03	84·970	5·490	2·290	28·610	10·240	36·980
Fæces	77·64	22·360	1·920	0·520	9·290	3·130	7·500
In hay eaten ..	..	3·144	0·203	0·085	1·059	0·379	1·368
In fæces excreted ..	..	1·267	0·109	0·030	0·526	0·177	0·425
Difference-digested ..	..	1·877	0·094	0·055	0·533	0·202	0·943
Percentage digestibility.	..	59·70	46·48	65·02	50·27	53·19	68·94
Mina.							
Bhusa 1923 [(wheat *bhusa*)	8·54	91·46	7·08	0·740	41·36	3·70	37·85
Dung	81·33	18·67	3·07	0·185	6·02	1·12	8·04
In *bhusa* given ..	..	116·60	9·03	0·944	52·73	4·72	48·26
In dung voided ..	..	45·36	7·46	0·450	14·62	2·72	19·54
Bhusa digested (by difference) ..	..	71·24	1·57	0·494	38·11	2·00	28·72
Digestibility coefficients.	..	61·10	17·38	52·32	72·26	42·36	59·51

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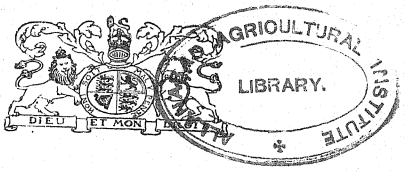
MEMOIRS OF THE DEPARTMENT OF AGRICULTURE IN INDIA

THE BUFFER ACTION OF SOME BURMA SOILS

BY

J. CHARLTON, M.Sc., A.I.C

Agricultural Chemist, Burma



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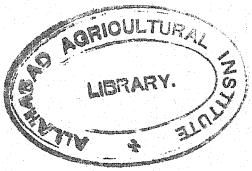

THE BUFFER ACTION OF SOME BURMA SOILS

BY

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Agricultural Chemist, Burma.

[Received for publication on the 7th April, 1924.]

I. INTRODUCTORY.

In rice producing countries such as Lower Burma where the rainfall averages ninety inches or more per annum, the annual loss of calcium carbonate from the soil is of great importance. It is well known that the majority of these soils are sour, and it is to be expected that they will gradually become more infertile for this reason alone, apart from other causes. The probable rate at which sourness will develop is, therefore, a very important matter.

Carbonates are, of course, not the only substances in the soil which resist the tendency towards sourness. Humus and other colloids resist change towards a more acid or more alkaline reaction, whereas carbonates resist only change towards an acid medium. Although, only substances resisting changes of reaction caused by both acids and alkalis can properly be called *buffers*, it is convenient to include carbonates because in general carbonates are the most important substances which prevent sourness in soils. To measure the amount of individual buffers in the soil is not possible because of the complexity of the problem. In the method developed, however, it is shown that useful information may be obtained by shaking soil with dilute acid and alkali and measuring the pH value of the soil solution so obtained. Arbitrary pH limits have been fixed at 4·5 and 9·5 and the behaviour of the soil examined between these limits.

Olof Arrhenius[1] obtained titration curves for certain soils, chiefly oriental. He did not deduce any expression for the *buffer action* of these soils. Buffer action or buffer effect may be defined as the rate of change of pH with addition

[1] Arrhenius, Olof. *Soil Science*, XIV, No. 3, pp. 223-232.

of acid and alkali. For equal additions of acid or alkali, soils showing a large buffer action will exhibit small changes of pH, while soils having a small buffer action will suffer relatively large changes of pH. Arrhenius found a general correlation between the titration curve and the fertility of the soil examined. A large buffer action usually indicated a good soil and small buffer action a poor soil.

Estimations of lime requirement such as that of Hutchinson and McLennan[1] are particular cases of the titration method in which the reaction of the soil is adjusted to a particular pH value. It has been shown by Warth and Po Saw[2] that the concentration of carbon dioxide in the calcium bicarbonate used causes large differences in the absorption of calcium carbonate, and they have developed a method of calculating lime requirement from the ratio $\frac{CaCO_3}{CO_2 \text{ in solution}}$ when equilibrium between soil and bicarbonate solution has been attained. For this purpose a typical neutral soil (Pwinbyu) was taken as standard. Further reference to this soil will be made subsequently.

In the method developed by Christensen[3] the growth or failure to grow of azotobacter demarked soils into two classes. In soils more alkaline than pH 5·9—6·0 azotobacter could grow whereas if more acid they failed to grow.

The advantages of titration curves as compared with actual lime requirements are apparent since some idea of the amount of buffers in the soil may be inferred from the curves.

II. EXPERIMENTAL.

To a series of flasks, each containing 20 grm. of air-dried soil which had passed the 3 mm. sieve, 100 c.c. of distilled water or 100 c.c. of dilute acid or alkali were added and the flasks closed with rubber stoppers. The flasks were then shaken in a shaking machine for a definite period at constant temperature. The soil solution was decanted, centrifugalised until clear and the pH value determined by Gillespie's method[4] using the comparator described by him. It was found that careful definition of the experimental conditions was necessary before consistent results could be obtained.

1. Acid and alkali used.

H_2SO_4 was used in all cases for the acid series. No objection to its use was found as it gave an extract which was always easily centrifugalised

[1] Hutchinson and McLennan. *Jour. Agri. Sci.*, 1915, 75.
[2] Warth, F. J., and Maung Po Saw. *Mem. Dept. Agri. India, Chem. Ser.*, Vol. V, No. 6.
[3] Christensen, H. R. *Tidsskr. Planteavl*, 1916, pp. 231–283.
[4] Gillespie, L. J. *Soil Science*, Vol. IX, No. 2, pp. 115–136.

to a clear solution. In the case of heavy calcareous soils, the addition of a drop or two of dilute H_2SO_4 is advantageous, since a clear extract is thereby more easily obtained and the pH value is altered only to a minute extent or not at all. The case of alkali is quite different. NaOH, Na_2CO_3, $Ba(OH)_2$, MgO, KOH and NH_4OH were used. Results varied according to the soil under examination. The cases of Pwinbyu, Palan and Padu soils will be considered.

(*a*) *Pwinbyu Soil.* Ten cubic centimetres each of N/10 Na_2CO_3, KOH, NaOH, $Ba(OH)_2$ and 0·02 grm. of solid MgO were each made up to a volume of 100 c.c. with distilled water and added to a series of flasks each containing twenty grams of air-dried Pwinbyu soil. The flasks were then shaken for twelve hours in a shaking machine at constant temperature (30°C.), the soil extract centrifugalised in corked vessels and the soil solutions examined. The MgO treated sample was quite clear, the Na_2CO_3 and KOH samples nearly so, while the NaOH and $Ba(OH)_2$ treated samples were slightly turbid.

(*b*) *Palan (Kyauktan) Soil.* Equivalent amounts of MgO and NaOH solution were tested against Palan soil in the same way as in the case of Pwinbyu soil. The soil solutions were shaken for half an hour and for two hours in each case after which the soil extracts were centrifugalised. The pH values obtained varied considerably although equivalent amounts of MgO and NaOH had been used (Table I).

TABLE I.

Palan (Kyauktan) soil. Behaviour with MgO and NaOH.

MgO			NaOH		
c.c. N/10 MgO added (as solid)	Reaction for		c.c. N/10 NaOH added	Reaction for	
	30 min.	2 hr.		30 min.	2 hr.
nil	5·7	5·70	nil	5·70	5·70
0·5	6·6	6·25	0·5	5·85	5·75
1·0	7·4	7·10	1·0	6·05	5·90
2·5	8·8	7·90	2·5	6·35	6·35
....	....	....	5·0	7·10	6·90

Results were as expected. Owing to the slight solubility of MgO, the time of reaction is very important. Whereas addition of nil to 5·0 c.c. N/10 NaOH gives practically the same pH value in either half an hour or two hours, in the case of 2·5 c.c. N/10 MgO there is a very marked difference. In case MgO is used the reaction must therefore be continued for a long period before equilibrium is obtained. It is not to be concluded that equilibrium is attained with NaOH in two hours. Such is not the case. The duration of reaction is treated later. In the present case of Palan soil the only inference is that in short periods different alkalis give different pH values.

(c) *Padu Subsoil.* Ten cubic centimetres each of N/10 NaOH, Na_2CO_3, $Ba(OH)_2$ KOH, NH_4OH and 0·02 grm. MgO were made up as before to a volume of 100 c.c. and added to a series of flasks each containing 20 grm. of Padu subsoil. The flasks were then shaken as in the previous cases but for twelve hours and the soil extracts obtained. In these soil extracts the pH values of only the MgO and $Ba(OH)_2$ treated samples could be read directly, all other extracts being very highly coloured largely owing to the presence of iron. Further, whereas in the case of NaOH the more alkali added, the deeper the colour of the soil solution obtained, in the case of MgO and $Ba(OH)_2$ the reverse is the case within wide pH limits. On adding sufficient MgO or $Ba(OH)_2$ to adjust the soil solution to pH 8·0—pH 9·0, water clear extracts were obtained.

The low solubility of MgO is a disadvantage for the practical part of the work and hence $Ba(OH)_2$ was selected as the most suitable alkali for all-round work. $Ba(OH)_2$ solution can be easily obtained at a strength of about 0·4 normal at ordinary temperatures and this is quite suitable for the work.

2. Duration of reaction with acid and alkali.

The only calcareous soil with which detailed experiments were made was Mandalay soil. This contains about one per cent. of carbonates of calcium and magnesium. It was found that the whole of the carbonates were not acted upon in less than about twelve hours at a constant temperature of 30°C., the soil and acid being constantly agitated in a shaking machine during the whole of this period. Tables II—V give the pH values of Mandalay, Pwinbyu, Hmawbi and Palan soils after reaction for thirty minutes, one, two, six, twelve and twenty-four hours respectively. The same results are plotted as titration curves in Charts I—IV showing only the half hour and twenty-four hour results for the sake of clearness. In all cases results are calculated to 100 grm. soil dried to constant weight at 100°C.

Table II.

Mandalay Farm Soil. pH value obtained with addition of H_2SO_4 and $Ba(OH)_2$. Reaction temperature 30°C. Results calculated to 100 grm. dry soil.

c.c. N.H_2SO_4 added	nil	0·53	1·32	2·64	5·28	7·92	10·56	13·20	15·84	18·48	21·12	23·76
30 minutes	8·05	7·95	7·60	7·2	6·45	5·75	4·90	<4·05	<4·05	..	..	..
1 hour ..	8·00	7·70	7·60	7·2	6·45	5·75	4·90	<4·05	<4·05	..	..	..
2 hours ..	8·05	7·85	7·65	7·4	6·60	6·05	5·35	4·70	<4·05	..	..	..
6 hours ..	8·05	..	..	..	7·20	6·50	5·90	5·70	5·10	4·6	<4·05	..
12 hours ..	8·05	..	..	..	..	6·80	6·35	6·05	5·65	5·0	4·60	4·25
24 hours ..	8·05	..	..	..	..	7·00	6·70	6·25	5·95	5·3	4·60	4·25

c.c. N.$Ba(OH)_2$ added	nil	0·96	1·11	2·4	2·78	4·8	5·55	7·2	8·33
30 minutes ..	8·05	8·4	..	9·4	..	>9·75	..	>9·75	..
1 hour ..	8·05	8·5	..	9·5	..	>9·75	..	>9·75	..
2 hours ..	8·05	8·5	..	9·5	..	>9·75	..	>9·75	..
6 hours ..	8·05	8·5	..	9·4	..	>9·75	..	>9·75	..
12 hours ..	8·10	8·3	..	9·0	..	9·55	..	>9·75	..
24 hours ..	8·05	..	8·3	..	9·0	..	9·5	..	>9·75

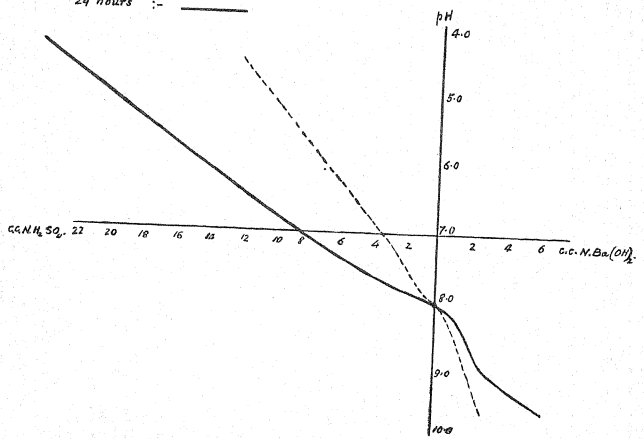


Chart I. Mandalay Soil.

Table III.

Pwinbyu Farm Soil. pH values obtained with addition of H_2SO_4 and $Ba(OH)_2$. Reaction temperature 30°C. Results calculated to 100 grm. dry soil.

c.c.N.H_2SO_4 added	nil	0·52	1·29	2·58	3·86	5·15	6·44	7·72	9·01	10·30
30 minutes ..	7·1	..	6·35	5·85	5·30	4·60	4·25	4·05	<4·05	..
1 hour ..	7·1	..	6·40	5·90	5·30	4·70	4·40	4·25	<4·05	..
2 hours ..	7·1	..	6·50	6·05	5·35	4·85	4·6	4·40	4·15	..
6 hours ..	7·2	..	..	6·25	5·85	5·50	4·85	4·50	4·15	<4·05
12 hours ..	7·1	..	..	6·25	5·85	5·30	4·90	4·70	4·45	4·20
24 hours ..	7·2	..	..	6·25	5·90	5·30	4·90	4·70	4·50	4·20

c.c.N.$Ba(OH)_2$ added	nil	0·99	2·47	4·95	7·42	9·89	12·36	14·845
30 minutes ..	7·0	7·2	7·6	8·50	9·3	9·55	>9·75	>9·75
1 hour ..	7·0	7·3	7·6	8·50	9·2	9·55	>9·75	>9·75
2 hours ..	7·0	7·4	7·7	8·40	9·3	>9·75	>9·75	>9·75
6 hours ..	7·0	7·4	7·7	8·30	9·2	>9·75	>9·75	>9·75
12 hours ..	7·0	7·4	7·6	8·30	8·9	9·50	>9·75	>9·75
24 hours ..	7·0	..	..	7·85	8·7	9·40	>9·75	>9·75

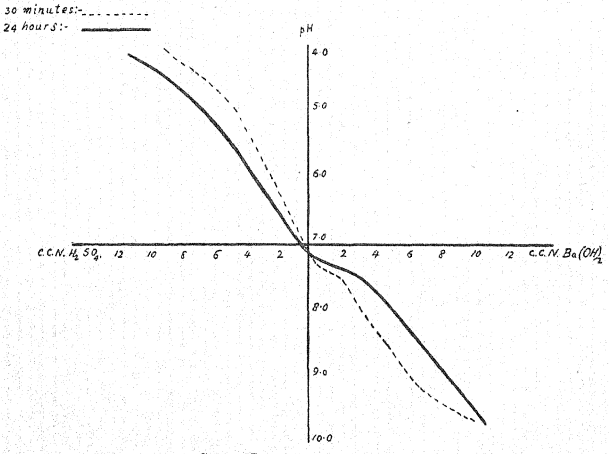


Chart II. Pwinbyu Farm Soil.

TABLE IV.

Hmawbi Farm Soil. pH values obtained with addition of H_2SO_4 and $Ba(OH)_2$. Reaction temperature 36°C. Results calculated to 100 grm. dry soil.

c.c. N.H_2SO_4 added	nil	0·13	0·26	0·51	1·28	2·56	3·85	5·01
30 minutes ..	6·05	6·05	5·40	4·9	4·40	4·05	<4·05	<4·05
1 hour ..	6·05	6·05	5·65	4·95	4·50	4·05	<4·05	<4·05
2 hours ..	6·05	6·05	5·55	5·1	4·50	4·20	<4·05	<4·05
6 hours ..	6·05	5·75	5·70	5·3	4·50	4·25	<4·05	<4·05
12 hours ..	6·05	6·05	5·70	5·3	4·60	4·25	<4·05	<4·05
24 hours ..	6·05	6·05	5·70	5·35	4·75	4·25	<4·05	<4·05

c.c.N.$Ba(OH)_2$ added	nil	2·46	2·85	5·70	5·93	7·39	8·54	9·85	11·14	12·31	14·24
30 minutes ..	6·15	6·8	..	..	7·60	8·50	..	9·2	..	>9·75	..
1 hour ..	6·15	6·7	..	..	7·60	8·40	..	9·2	..	>9·75	..
2 hours ..	6·15	6·7	..	..	7·60	8·40	..	9·0	..	9·55	..
6 hours ..	6·15	6·7	..	..	7·50	8·20	..	8·9	..	9·50	..
12 hours ..	5·95	6·7	..	..	7·45	8·15	..	8·5	..	9·50	..
24 hours ..	6·05	..	6·9	7·65	..	..	8·2	..	9·2	..	>9·75

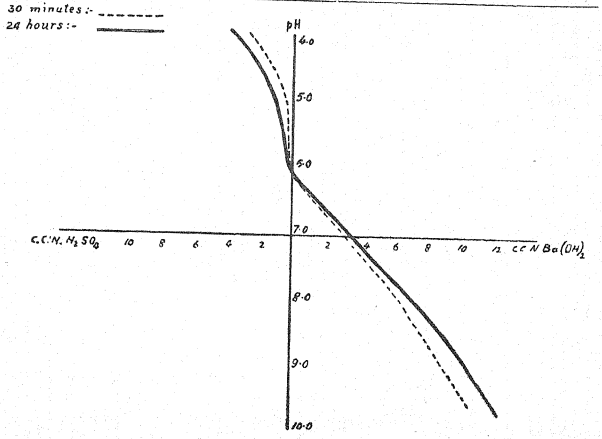


CHART III. Hmawbi Farm Soil.

TABLE V.

Palan (Kyauktan) Soil. pH values obtained with addition of H_2SO_4 and $Ba(OH)_2$. Reaction temperature 30°C. Results calculated to 100 grm. dry soil.

c.c. N.H_2SO_4 added	nil	0·13	0·27	0·54	1·34	2·68	4·02
30 minutes ..	5·75	5·55	5·3	4·6	4·05	<4·05	..
1 hour ..	5·75	5·55	5·2	4·8	4·25	<4·05	<4·05
2 hours ..	5·80	5·55	5·3	4·8	4·25	4·05	<4·05
6 hours ..	5·75	5·55	5·3	4·9	4·50	4·30	<4·05
12 hours ..	5·80	5·55	5·3	5·0	4·50	4·20	<4·05
24 hours ..	5·80	5·55	5·3	4·9	4·40	4·10	<4·05

c.c. N.$Ba(OH)_2$ added	nil	2·57	2·97	5·14	5·95	7·71	8·92	10·28	11·89	12·85	14·86	15·42
30 minutes ..	5·8	6·40	..	7·2	..	7·95	..	8·70	..	9·35	..	>9·75
1 hour ..	5·7	6·40	..	7·0	..	7·80	..	8·20	..	9·40	..	>9·75
2 hours ..	5·8	6·40	..	7·1	..	7·85	..	8·60	..	9·50	..	>9·75
6 hours ..	5·8	6·35	..	7·0	..	7·70	..	8·50	..	9·30	..	>9·75
12 hours ..	5·7	6·25	..	7·0	..	7·70	..	8·35	..	9·30	..	>9·75
24 hours ..	5·8	..	6·35	..	7·2	..	7·85	..	8·9	..	9·75	..

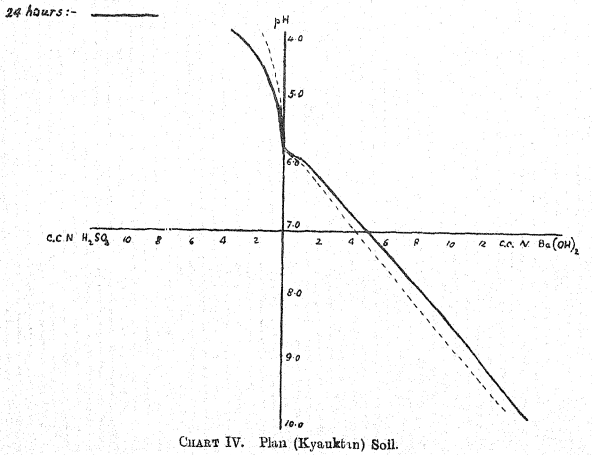


CHART IV. Plan (Kyauktan) Soil.

In drawing in the titration curves the general direction was followed. In this way it is possible that buffered action over small pH ranges is not shown.

From a consideration of Tables II—V and Charts I—IV it is clear that

(*a*) In the case of calcareous soils even when these do not contain more than one per cent. $CaCO_3$, equilibrium with dilute H_2SO_4 is not attained in less than 24 hours. It is probably not attained even in that period but the absorption of H_2SO_4 in about 12 hours is greater than that demanded by the presence of one per cent. $CaCO_3$ alone since to decompose one gram of $CaCO_3$ about 20 c.c. of N.H_2SO_4 are sufficient. Differences in the absorption of $Ba(OH)_2$ are small over a comparatively large time interval.

(*b*) In the case of neutral and sour soils, absorption of both H_2SO_4 and $Ba(OH)_2$ differ little even after reaction times so widely different as 30 minutes and 24 hours. The greater difference occurs in the case of the neutral soil both for acid and alkali.

It therefore appears advisable to continue the reaction for a period of 24 hours in all cases, although with soils which are known to be sour, very little error is introduced even if the reaction is allowed to proceed for 30 minutes only.

3. Temperature of reaction.

Detailed experiments to determine the difference due to changes of temperature of reaction were not attempted. Any difficulty due to temperature changes was avoided by constructing a wooden box lined with asbestos and fitting the same round the shaking machine. The temperature in all experiments was kept constant at 30°C. $\pm$ 1°C., *i.e.*, the lowest temperature which can be conveniently used in Mandalay.

4. Precautions necessary when using $Ba(OH)_2$ solution.

When measuring $Ba(OH)_2$ solution from a pipette it was found very necessary to avoid blowing down the pipette in the slightest degree, otherwise precipitation of $BaCO_3$ occurred. Errors due to this cause undoubtedly occurred in the earliest experiments but not in those quoted in this paper. Provided that care was observed, no precipitation of $BaCO_3$ occurred. Further, in all experiments quoted, the exposure of the soil solutions treated with $Ba(OH)_2$ was reduced to a minimum and while centrifugalising, the bottles were closed with rubber stoppers.

5. Dilution of soil solutions.

Theoretical evidence has been given to show that dilution of the soil solution may be practised without seriously altering the pH value obtained.[1] Dilution with distilled water both freshly boiled and otherwise was tried on certain soil solutions to confirm this (Table VI).

Table VI.

Dilution of soil solutions.

	pH Value					
Undiluted soil extracts	5·70	6·35	7·00	7·95	8·4	8·9
Extracts diluted with equal volume of distilled water of pH 5·4 ..	5·50	5·95	6·15	6·60	6·8	6·90
Extracts diluted with equal volume of distilled water of pH 7·0 ..	5·75	6·50	7·10	8·00	8·3	8·70
Extracts diluted with equal volume of distilled water of pH >9·75 ..	7·00	7·30	7·90	8·70	9·5	>9·75

At the time of the experiment, a sample of ordinary distilled water in the laboratory gave a pH=5·4; one that had been gently boiled for about five minutes and then quickly cooled gave a pH=6·9—7·0, while one that had been briskly boiled for thirty minutes and had then been quickly cooled gave a pH>9·75. This was due to solution of soda from the glass by the water. Unfortunately all the large glass vessels in the laboratory were of the same manufacture and hence the method of diluting turbid or highly coloured soil solutions for determination of the pH value could not be carried out. A centrifugal machine holding eight tubes each of 100 c.c. capacity was used to clear the turbid soil solutions and the pH values were determined direct. The centrifugal machine revolved at 2,000–2,400 revolutions per minute, and it was found that even heavy clay soils could be sufficiently cleared for the determination in a maximum of two hours' centrifugalising.

6. Recommended method.

To a series of flasks, usually eight, each containing twenty grams of air-dried fine soil which have passed the 3 mm. sieve, *i.e.*, soil for mechanical

[1] Sharp, L. T., and Hoagland, D. R. *Jour. Agri. Res.*, Vol. VII, p. 124.

analysis, 100 c.c. of dilute H_2SO_4 or $Ba(OH)_2$ are added in increasing concentration to seven and 100 c.c. of distilled water to the eighth. The flasks are closed with rubber stoppers and placed in a shaking machine with constant temperature device and shaken for twenty-four hours at 30°C. The soil solution is then decanted into centrifugal tubes which are thereupon closed with rubber stoppers and centrifugalised until clear. After decanting the clear solutions into clean dry vessels, the pH values are determined by Gillespie's method using a comparator to obviate difficulties due to slight colour or slight turbidity.

It is necessarily assumed that H_2SO_4 and $Ba(OH)_2$ behave similarly to other acids and alkalis respectively. This is possibly by no means the case. In particular the reaction with alkalis may be expected to differ since carbonates of sodium, potassium and ammonium are very soluble, whereas $CaCO_3$ and $BaCO_3$ are practically insoluble. Lime requirements made by using $Ba(OH)_2$ should, however, be comparable with those determined by using $Ca(HCO_3)_2$ since the solubilities of $BaCO_3$ and $CaCO_3$ are both very small and of the same order. Whether justifiable or not, this has been assumed largely because of the convenience of using $Ba(OH)_2$ solution.

III. NUMERICAL VALUES FOR BUFFER ACTION APPLIED TO BURMA SOILS.

G. Lehmann[1] has measured the buffer action of physiological solutions by means of the expression

$$\frac{b}{pH_1 - pH_2}$$

where pH_1 was the original and pH_2 the pH developed after adding a small quantity b of N/100 acid or alkali to ten cubic centimetres of the solution. For such solutions a buffer action of 8·0 indicated a strong, 0·5 to 4·0 a weak and 0·5 or less no buffer action. Blood gave a buffer action of 11·0.

The case of soil solutions is very different from that of physiological solutions in which only small changes of pH are usually measured. In the first place it has been assumed that the range of fertility of soils is from pH 4·5 to pH 9·5. A mathematical expression for the buffer action of a soil should fulfil the following :—

(*a*) The relative amounts of acid and alkali required to alter a soil from its existing pH value to pH 4·5 and pH 9·5 respectively should be shown.

[1] Lehmann, G. *Biochem. Zeits.*, 1922, 133, 30—45.

(*b*) There must be no possibility of confusing well buffered and poorly buffered soils. Hence the sum and not the difference of acid and alkali used must be shown.

(*c*) The difference between very sour and very alkaline soils must be indicated.

(*d*) It is undesirable to show absorption of acid and alkali as buffer action when it is known to be due to presence of free acids or free alkali. This condition is shown to some extent by the existing pH of the soil.

No simple algebraic expression fulfils the above conditions. It is therefore better to confine the problem to a simple issue. This is done by expressing the buffer action against sourness and alkalinity separately.

E.g. Buffer action against sourness $= \frac{\text{c.c.N.H}_2\text{SO}_4 \text{ used}}{\text{X} - 4{\cdot}5}$ per 100 grm. dry soil where X is the existing pH value of the soil. In the same way, the buffer action against alkalinity $= \frac{\text{c.c.N.Ba (OH)}_2 \text{ used}}{9{\cdot}5 - \text{X}}$ per 100 grm. dry soil where X is the existing pH value of the soil.

Under actual conditions the above method is justifiable since soils are becoming either more sour or more alkaline and trouble from one cause or the other, not both, may be anticipated. In the case of Lower Burma paddy soil the trouble is that of increasing sourness. For these soils, therefore, the value of the expression $\frac{\text{c.c.N.H}_2\text{SO}_4 \text{ used}}{\text{X} - 4{\cdot}5}$ indicates the type of soil but the total number of cubic centimetres of $N.H_2SO_4$ required to adjust the reaction of the soil from its existing pH value to pH 4·5 is a direct measure of the reserve fertility of the soil against sourness. In Table VII a list of Burma soils is given showing the buffer action against $N.H_2SO_4$ and $N.Ba(OH)_2$ per 100 grm. dry soil together with other data. Larger scale curves than those in Charts I—VII were drawn to determine these data more accurately.

The Lower Burma paddy soils quoted in Table VII are Hmawbi, Palan, Kyeinkagon, Kungyaunywa, Letpangin, Sinyokin and Magyibin, but the Akyab and Pyinmana soils may be considered along with these since they grow paddy under rainfall without irrigation. The Tonkan Forest Reserve soil is of the same type as Kyeinkagon and Letpangin and at some future date may grow paddy since this forest reserve is in the plains.

The total buffer action against $N.H_2SO_4$ shows that the Palan soil will probably be the first to become infertile through sourness (1·15), followed by Hmawbi (1·85) and Akyab waterworks (1·90). The most productive soils

TABLE VII.

Buffer action values of Burma soils.

Soil	Actual pH	Lime requirement per cent. from titration curves	Total buffer action against $N.H_2SO_4$	Buffer action against $N.H_2SO_4$ per 1·0 pH	Total buffer action against $N.Ba(OH)_2$	Buffer action against $N.Ba(OH)_2$ per 1·0 pH	Annual rainfall (Approximate)	REMARKS
1. Mandalay ..	8·05	nil	22·50	6·34	5·50	3·80	....	Irrigated paddy calcareous water.
2. Pwinbyu ..	7·10	nil	9·00	3·46	10·00	4·17	....	Irrigated paddy calcareous water.
3. Hmawbi	6·05	0·1650	1·85	1·19	12·30	3·57	95″	Paddy under rainfall.
4. Palan	5·75	0·2600	1·15	0·92	14·10	3·76	112″	Paddy under rainfall.
5. Tatkon soil ..	6·90	0·0125	4·25	1·77	9·00	3·46	40″	All crops except paddy.
„ subsoil ..	6·90	0·0125	3·55	1·48	8·60	3·31		
6. Akyab—								
Waterworks soil ..	6·50	0·0350	1·90	0·95	5·85	1·95	100 to 225″	Paddy under rainfall.
Waterworks subsoil	7·15	nil	3·20	1·21	5·25	2·23		
Mingan soil ..	5·85	0·1300	2·45	1·81	13·85	3·80		
Mingan subsoil ..	6·45	0·0575	3·25	1·67	13·60	4·46		

TABLE VII—*contd.*

Soil	Actual pH	Lime requirement per cent. from titration curves	Total buffer action against $N.H_2SO_4$	Buffer action against $N.H_2SO_4$ per 1·0 pH	Total buffer action against $N.Ba(OH)_2$	Buffer action against $N.Ba(OH)_2$ per 1·0 pH	Annual rainfall (Approximate)	REMARKS
7. Pyinmana—								
Highest land soil ..	6·50	0·0950	3·90	1·95	13·20	4·40	50″– 55″	Paddy under rainfall, also sugarcane.
Highest land subsoil	6·35	0·1150	3·25	1·76	13·30	4·22		
Lowest land soil ..	6·45	0·0500	3·65	1·87	9·20	3·02		
Lowest land subsoil	6·50	0·0700	3·75	1·88	9·20	3·07		
8. Tonkan—								
Forest Reserve soil..	6·35	0·1325	4·50	2·43	16·30	5·18	130″–140″ ?	Open teak forest (Plains).
Subsoil ..	6·10	0·2000	3·50	2·19	18·50	5·44		
9. Kyeinkagon—								
Soil	6·55	0·1100	6·40	3·12	14·90	5·05	130″–140″ ?	Medium to long lived paddies under rainfall.
Subsoil ..	6·80	0·0300	6·40	2·78	16·20	6·00		
10. Kungyaunywa—								
Soil	6·10	0·2500	5·00	3·13	34·80	10·23	130″–140″ ?	Medium to long lived paddy. District subject to floods.
Subsoil ..	6·60	0·0600	5·60	2·67	24·20	8·35		

11. Letpangin—								
Soil ..	..	6·60	0·0550	6·30	3·00	20·10	6·93	130″–140″ ? Long lived paddy.
Subsoil	..	6·40	0·1500	3·90	2·05	17·80	5·74	
12. Sinyokin—								
Soil ..	..	6·20	0·1750	3·20	1·88	17·60	5·33	130″–140″ ? Medium lived paddy. Very sandy soil.
Subsoil	..	6·35	0·1250	2·70	1·46	13·80	4·38	
13. Magyibin—								
Soil ..	..	6·15	0·2900	4·60	2·79	21·20	6·33	130″–140″ ? Medium paddies. Soil not very fertile.
Subsoil	..	6·55	0·1250	5·00	2·44	19·60	6·64	

are the five last named soils in Table VII which have a total buffer action of 3·2 to 6·4. These five soils are subject to flood from the Sittang river and its tributaries but when flooding is not too serious, all these soils yield excellently and are among the best paddy soils in Burma. The low buffer action of Hmawbi and Palan is attributable to the fact that they are on the old alluvium and the rainfall is very heavy. There is indeed a rough but imperfect correlation between the total buffer action against $N.H_2SO_4$ and the rainfall. The greater the rainfall the smaller the buffer action against $N.H_2SO_4$.

The average of the non-calcareous paddy soils growing paddy under rainfall quoted in Table VII gives the following results, subsoils not being included:

Buffer action against $N.H_2SO_4$ per 1·0 pH = 2·05.
„ „ „ $N.Ba(OH)_2$ „ „ „ = 4·94.

Although insufficient samples have yet been treated to generalize, it appears that the best non-calcareous paddy soils should have a buffer action against $N.H_2SO_4$ equal to not less than 3·0 per 1·0 pH. The value of the buffer action against $Ba(OH)_2$, both total and per unit pH does not seem so important except that it indicates greater leaching and therefore greater lime requirement. The extremely high values for the Kungyaunywa soils are probably connected with the prevalence of floods in that district. In soils of the same type there is a rough correlation between the mechanical texture of the soil and the buffer action against $N.Ba(OH)_2$ per unit pH. Thus in the case of the Tonkan Forest Reserve, Kyeinkagon, Kungyaunywa, Letpangin, Sinyokin and Magyibin soils, the heaviest soils, Kungyaunywa and Magyibin have larger than average buffer action against $Ba(OH)_2$. Mechanical analyses of soils are given in Table VIII. In Table VIII the soil type has been determined by adding together the fractions Clay + Fine Silt, Silt + Fine Sand, Coarse Sand + Fine Gravel and plotting the results within an equilateral triangle divided into six triangles of equal size by joining the apices of the equilateral triangle to the midpoints of opposite sides. In this way six equal triangles were obtained corresponding to soils of varying degree from the heaviest clays to the coarsest sands. The soils have been numbered consecutively from one to six in order of decreasing fineness of texture, *i.e.*, number 1 is the heaviest clay and number 6 the coarsest sand.

With regard to soils other than Lower Burma paddy soils quoted, Mandalay is a heavy calcareous clay. Pwinbyu is a well balanced clay by analysis of ultimate particles. Actually it contains sufficient organic matter to render it moderately free working. Palan and Hmawbi are typical Delta clay soils on the old alluvium. Pyinmana is in the intermediate zone and hence although

the soils are of the loam type the buffer action is fair. Tatkon soil (Chart V) is very similar to that of Pyinmana although lighter than the latter. Tatkon is on the edge of the dry and intermediate zones. The Akyab waterworks soil is the coarsest soil growing paddy that the author has hitherto discovered. Although the rainfall is upwards of 200″ per annum, this would not retain moisture sufficiently well to grow paddy were it not for the fact, that this soil

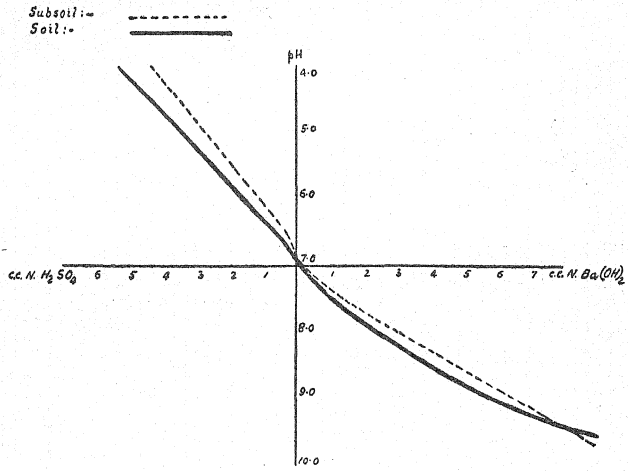


CHART V. Tatkon Farm Soil.

derives an underground supply of water from a reservoir close by. The Mingan soil is a more normal paddy soil but poorly buffered against H_2SO_4 probably on account of the high rainfall.

There is much land of the Palan type which is now yielding only about 1,000 lb. of grain per acre in Lower Burma. It is a noteworthy fact that on the paddy soils Kyeinkagon, Kungyaunywa, Letpangin, Sinyokin and Magyibin a large proportion of the paddy crop becomes laid, particularly a medium lived

TABLE VIII.

Mechanical Analysis of Burma Soils.

	Mandalay	Pwinbyu	Hmawbi	Palan	Tatron		Akyab				Fyinmana			
							Waterworks		Mingan		Highest land		Lowest land	
					Soil	Subsoil	Soil	Subsoil	Soil	Subsoil	Soil	Subsoil	Soil	Subsoil
Stones and gravel ..	nil	0·01	nil	1·97	0·03	..	0·04	0·46	0·18	0·28	nil	nil	nil	nil
Fine gravel	0·92	0·13	nil	8·05	0·22	..	1·51	..	0·56	2·30	nil	nil	nil	nil
Coarse sand	6·08	0·78	0·73	..	32·28	..	48·37	53·97	0·31	2·50	9·6	10·0	11·6	9·0
Fine sand ..	17·25	12·60	6·67	9·36	35·29	..	16·15	15·08	2·94	2·45	31·0	34·1	42·3	45·2
Silt ..	13·29	35·07	17·44	23·42	13·72	..	12·87	10·45	37·27	33·00	25·5	23·9	21·5	26·6
Fine Silt ..	18·16	31·92	41·25	17·52	9·38	..	13·75	16·94	35·79	34·32	16·2	21·3	15·3	18·1
Clay ..	44·30	19·49	33·91	39·68	9·08	..	6·60	2·86	22·16	24·53	15·9	10·6	9·5	5·3
Type No. ..	1	1	1	1	4	..	6	6	1	1	3	3	3	3
Description	Clay	Clay	Clay	Clay	Light Loam	..	Coarse sand	Coarse sand	Clay	Clay	Loam	Loam	Loam	Loam

TABLE VIII—*contd.*

	Tonkan Forest Reserve		Kyeinkagon		Kungyaunywa		Letpangin		Sinyokin		Magyibin	
	Soil	Subsoil	Soil	Subsoil	Soil	Subsoil	Soil	Subsoil	Soil	Subsoil	Soil	Subsoil
Stones and gravel ..	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil
Fine gravel	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil
Coarse sand	10·6	7·3	16·6	4·2	0·3	0·4	5·2	4·8	14·8	17·1	1·9	1·9
Fine sand	32·9	27·9	36·7	45·1	10·8	6·5	36·1	43·1	42·7	44·0	7·7	10·9
Silt	25·8	26·1	24·0	23·6	36·8	36·0	21·7	21·5	18·8	19·1	35·6	35·6
Fine silt	22·3	28·7	21·1	21·4	41·1	45·2	25·6	21·5	18·0	15·3	36·7	38·7
Clay	9·1	10·4	4·9	7·4	12·2	13·4	9·2	7·3	7·7	6·4	15·8	11·9
Type No.	3	3	3	3	1	1	3	3	3	3	1	1
Description	Loam	Loam	Loam	Loam	Clay	Clay	Loam	Loam	Loam	Loam	Clay	Clay

paddy called Letywezin. Whether this is due to heavy yield, the actual sourness of the soil or the difficulty experienced by the plant in procuring an adequate supply of calcium is an interesting problem and it has been suggested by the author that experiments with lime should be tried. Letywezin is grown only on the sour soils of Lower Burma and is much esteemed as a fair or good yielder.

It is possible to make an interesting comparison between calcareous and non-calcareous soils by estimating the carbonates present in the former and deducting the $N.H_2SO_4$ used in decomposing this carbonate. On this basis the buffer action against $N.H_2SO_4$ per unit pH of the Mandalay soil is only 0·79, the lowest for all the soils quoted. This probably indicates that the reaction with dilute H_2SO_4 is not complete in twenty-four hours but it may indicate that the Mandalay soil lacks an adequate supply of organic matter.

In general the buffer action against $N.H_2SO_4$ per unit pH is greater in the soil than in the subsoil, the case of Akyab soils being an exception. Where both soil and subsoil are free from $CaCO_3$ and the soil does not contain more colloidal clay than the subsoil, the greater buffer action of the soil may be attributed to its higher content of organic matter. This suggests a method of amelioration of soils poorly buffered against sourness, *viz.*, by green manuring, although better results would probably be obtained by liming in conjunction with green manuring.

IV. CONCLUSIONS.

1. The range of fertility of soils is assumed to lie between the limits pH 4·5 and pH 9·5.

2. The buffer action of soils against sourness and alkalinity may be determined by shaking soil for twenty-four hours at 30°C. with H_2SO_4 or $Ba(OH)_2$ solution respectively and measuring the pH values of the soil solutions obtained. The centrifugal method of clearing the solutions is preferred. From the pH values obtained, titration curves are plotted.

3. From the titration curves, the lime requirement of a soil may be read at any particular pH value. By dividing the number of cubic centimetres of $N.Ba(OH)_2$ absorbed by twenty, the lime requirement per cent. for adjusting the reaction of the soil to the particular pH value required is obtained.

4. A measure of the total buffer action against increasing sourness or increasing alkalinity is given by the number of cubic centimetres of $N.H_2SO_4$ or $N.Ba(OH)_2$ respectively required to transform the reaction of the soil from its existing value to pH 4·5 and pH 9·5, results being expressed per 100 grm. soil dried to constant weight at 100°C.

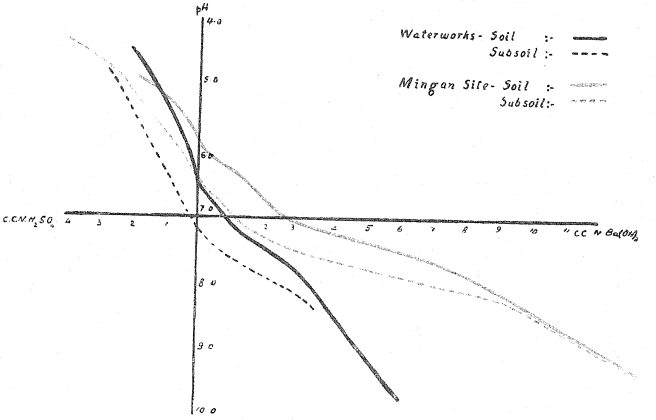


Chart VI. Akyab Soils.

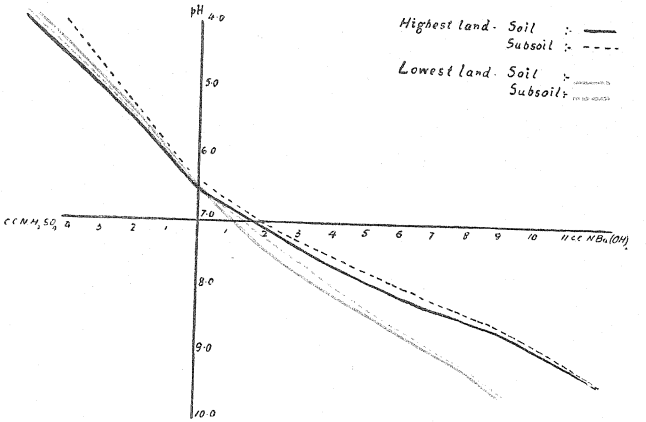
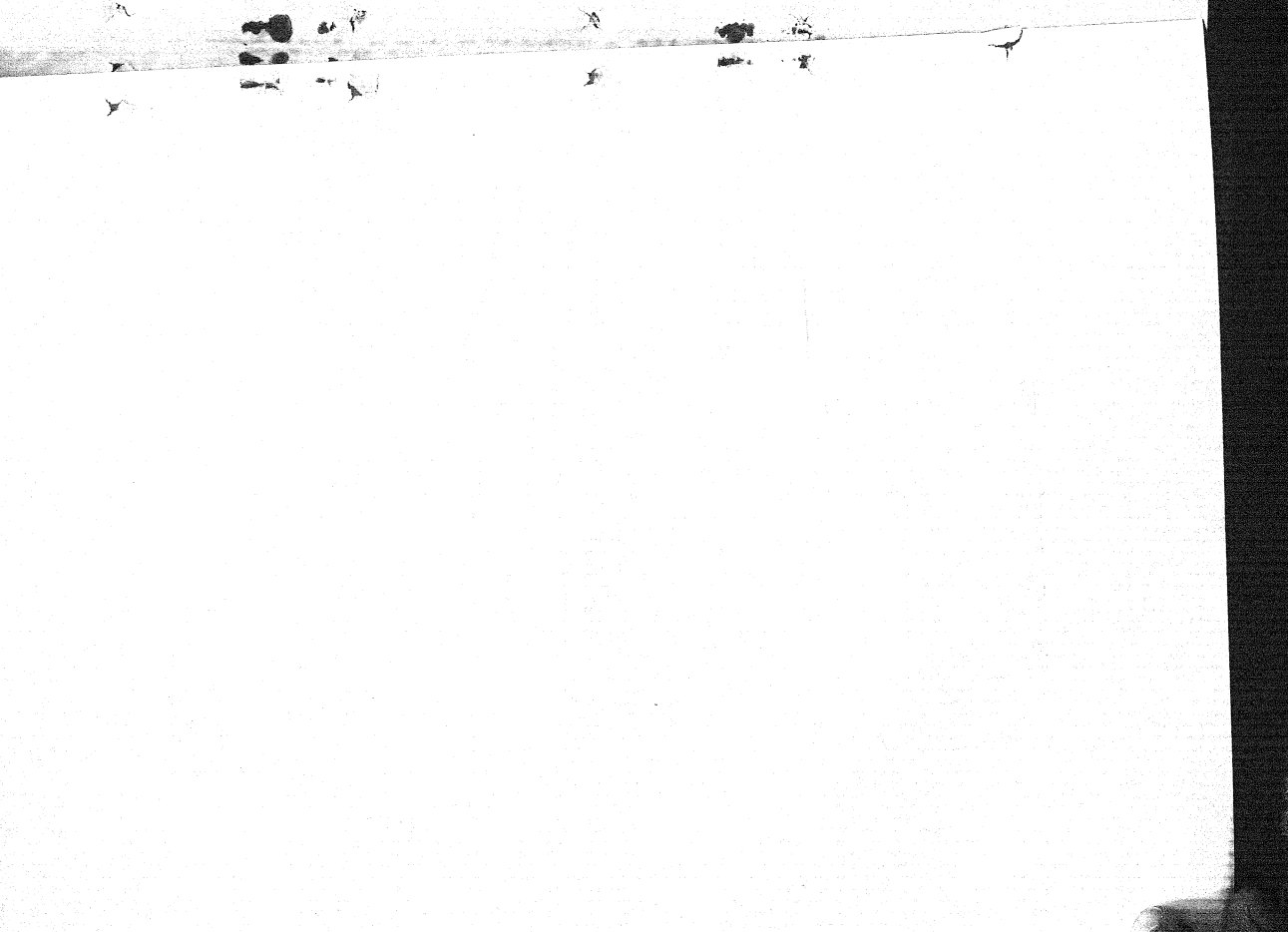


Chart VII. Pyinmana Farm Soils.



5. The buffer action of soils against acid and alkali respectively may be expressed as

$$(a)\ \frac{\text{c.c. N.H}_2\text{SO}_4 \text{ used}}{X-4{\cdot}5}$$

$$(b)\ \frac{\text{c.c. N.Ba(OH)}_2 \text{ used}}{9{\cdot}5-X}$$

where X is the existing pH value of the soil and the weight of soil used is equal to 100 grm. dried to constant weight at 100°C.

6. A selection of non-calcareous Burma soils growing paddy exclusively under rainfall gives a value for buffer action against acid per unit of pH varying from 0·92 to 3·42 with a mean of 2·04. The better soils have the higher values. Sour paddy soils should have a value of not less than 3·0.

7. Buffer action against alkali is relatively unimportant in the soils examined. In the non-calcareous paddy soils the average value per unit of pH was 4·94, the more productive soils having the higher value. The only inference which can be drawn with any certainty in such cases is that the higher the buffer action against alkali, the greater the lime requirement over certain pH ranges.

The author desires to acknowledge his indebtedness to Mr. S. P. Aiyar, B.A., who has determined all the pH values of the soils quoted except such as were necessary in standardizing the method adopted.

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MEMOIRS OF THE DEPARTMENT OF AGRICULTURE IN INDIA

STUDIES IN THE CHEMISTRY OF SUGARCANE, II

SOME FACTORS THAT DETERMINE THE RIPENESS OF SUGARCANE

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STUDIES IN THE CHEMISTRY OF SUGARCANE, II.*

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BY

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[Received for publication on 20th May, 1924.]

In a previous paper[1] submitted to the Bombay session of the Science Congress (1919) by one of us, the determination of the ripeness of sugarcane was one of the questions sought to be investigated. The paper dealt with the preliminaries of the problem, and the experiments recorded therein aimed at finding out the inter-relations of one internode to another of the same cane with the ultimate object of basing some method of determining the ripeness of a cane on such relations. It has been shown that when the cane is young and consequently immature, the difference in the contents of total solids calculated as sucrose between a bottom internode and a top internode is very great and in favour of the one below, and as the cane becomes older and more mature this difference tends to disappear and even gets reversed in favour of the internode at the top.

The work carried out subsequent to the year 1919 with a number of varieties of canes and in different seasons, besides confirming the observations

* Paper read at the Indian Science Congress, Bangalore, 1924.

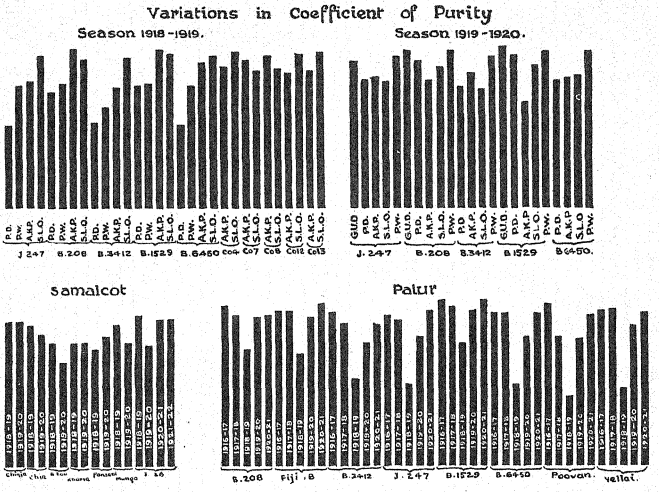
[1] *Agri. Jour. India*, XIV, 3.

previously recorded, brings prominently to our view certain factors which have a bearing on the determination of ripeness of sugarcane by chemical methods. The present paper is a record of these experiments and observations, and attempts at the improvement of the existing methods of chemical analysis of sugarcane.

Before presenting the subject proper it is essential to examine critically the methods that now obtain for determining the ripeness of a cane. The usual method is to determine the coefficient of purity of the juice of the cane under question, and when this coefficient reaches a certain value which is different for different varieties, the cane is declared to be ripe. In the case, however, of manurial experiments or varietal tests with new canes or seedlings a more detailed information is sought for by carrying out periodical analysis. Whether the test is one involving a single or many periodical analyses, the coefficient of purity has been the main guiding factor in judging when a cane is ripe, that is, fit for harvest either for *jaggery* (raw, crude sugar) or for sugar-making. The coefficient of purity differs with varieties. This is certainly a serious disadvantage, for the method becomes practically useless when the analyst is not acquainted with the variety. Still again for the same variety the coefficient is different in different places and soils. In other words, conditions of soil, climate, manuring, and irrigation affect the value of the coefficient to a very appreciable extent. In Tables I–IV will be found recorded the variations in the values of the coefficients found in different places for the same season and variety and for the same variety and place in different seasons. These figures are also shown graphically in Chart I. These coefficients were obtained from the results of analyses of canes analysed at a time when, in the opinion of the agricultural officers in charge of the experiment stations, they were considered ripe and fit for harvest. It will be found from the tables that differences of 5 to 10 per cent. in the coefficients between one place and another are quite common. Greater differences also occur though not so commonly. Palur figures for wet and dry lands for 1919-1920 are significant. Here the influence of soil conditions alone is exemplified. Similar differences are also noticeable for coefficients for various seasons in the same place. In Table IV(*a*) are recorded the variations in purity alongside variations in the total solid content. It will be found that differences much smaller than 5 per cent. correspond to a considerable increase in the total solid content and are therefore of no small importance in judging if a cane is ripe or not. This, taken in conjunction with the large variations detailed in Tables I–IV, clearly shows that the purity is not always a reliable factor for judging the maturity of a cane; especially when dealing with a cane the

nature or history of which one has no knowledge. For instance, if a cane like B ·208 which is reputed to give a juice of about 95 per cent. purity at the usual ripening season gives only a purity of about 80 to 85 per cent., it would

CHART I.



KEY.

P. D. = Palur dry land.
P. W. = Palur wet land.
A. K. P. = Anakapalle.
S. L. O. = Samalcot.
G. U. D. = Gudiyattam.

be difficult to say whether the crop is ripe or not. The crop may not have come to the ripening state although it is about 11 or 12 months old, or the capacity of the crop is only that much ; which of them is the correct estimate we are not able to say from the data before us.

Migake Ishida[1] of the sugar experiment station, Formosa, has put forward another method of determining the ripeness of sugarcane. The following is the

[1] *Bull. Govt. Sugar. Expt. St., Formosa, Japan*, No. 1, pp. 6–7.

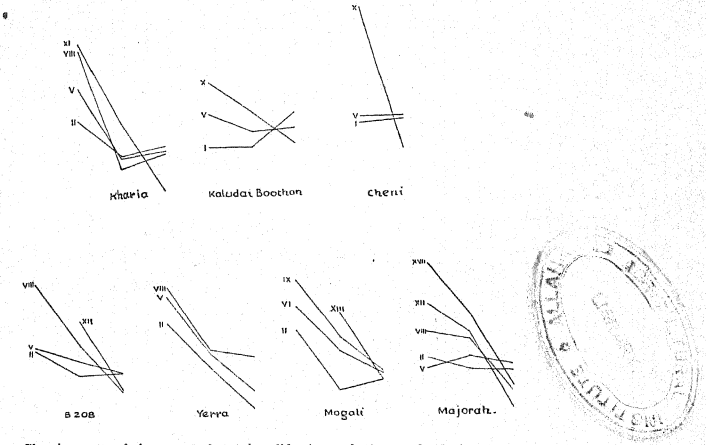
method as described by the writer himself. He says, " In the study of the maturity of the sugarcane the writer has proposed a new criterion which has been called the maturity coefficient. It consists in finding the ratio of reducing sugar to sucrose . . . . In experiments which have been pursued for several years upon many varieties of sugarcane it has been ascertained the period in which any variety of sugarcane makes a great change of its maturity coefficient, this period occurs at almost the same season every year and shows about the same value of coefficient, if the sugarcane, of course, is planted in the same season and other treatments are the same."

The results obtained by this author with a variety of cane known as Rose Bamboo are extracted in Table V for reference. Fuller details are not available, but from these two sets of figures, no doubt put forward by him as typical ones, it will be found that the new ratio is quite as unreliable as the old one. Like the coefficient of purity, the maturity coefficient also varies considerably. For instance, for the same variety of cane the maturity coefficient varied between 256·79 and 79·9 between the years 1912-13 and 1913-14. Again in the figures for 1913-14 there are two periods marked by the large increase in maturity coefficient referred to. During the fortnight between 26th December and 9th January the coefficient rises from 15·4 to 71·8; but during the two fortnights that follow 9th January it falls again almost to the original figure of 26th December to rise again on 6th March to 79·9. Besides the ratio is admittedly dependent for its value and period on temperature, soil conditions and methods of treatment. In the absence of additional information we cannot say anything definite about the method but, so far as available evidence goes, the method has nothing to recommend it in preference to the older one.

It is thus seen that the coefficient of purity or maturity are variable factors and therefore not always reliable for determining whether a cane is mature. It is natural that it should be so depending as it does on the sucrose content on the one hand and the other soluble substances of cane-juice on the other. In its place we have to search for a factor which can be more independent of the internal changes and adjustments of the cane. It is this consideration that prompted us to place more faith on the total solid content of the juice than on its sucrose or glucose contents, or any inter-relations between them. In the paper referred to in the beginning, it has been shown that, as the cane grows, there is a general levelling about the middle of the curves indicating either the possibility of the sugar moving upwards or its being used up in the formation of cane tissue. This fact presented in a slightly different light leads on to the conclusion we have arrived at, regarding the maturity of

a cane. In Table VI are given the results of examination of a few select internodes of a cane side by side with the increase of sugars in each internode between any two intervals. These increases are plotted against intervals for each cane in Chart II. It should be noted that these are the results of periodical examination of single canes while standing in the field according to the method detailed in the paper by one of us already referred to. It will be found that, during the early stages, the curves for the top internodes

CHART II.



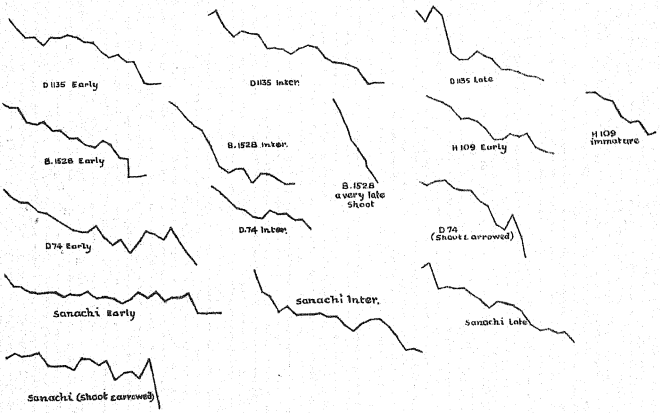
Showing rate of increase of total solids in each internode during intervals of a month.
Roman figures indicate the number of the internode from the bottom.
Ordinates = per cent. increase in total solids.
Abscissae = intervals.

stand always above those of the bottom ones. In other words, as the cane matures, whatever might be the fate of the sugars, whether they move upwards to fill up younger internodes or are used up in the building up of cane tissue, the general tendency is to equalize all differences in total-solid-contents between the various internodes up to the highest dead leaf joint. Conversely, when the total-solid-contents of the bottom and top internodes are equal or

nearly equal, i.e., when their ratio is unity or nearly unity, the cane may be declared to be ripe.

With the object of further confirming our hypothesis, about the latter half of April 1921, a dozen canes were marked out with botanical descriptions. Some of them were labelled as early or mature, some as intermediate, or maturing, while others were described as late or decidedly immature canes as judged by the eye. Each of these was examined, internode after internode,

CHART III.



Showing the shape of curves of canes of different age as judged by botanists.

for their total-solid-content by the refractometer. It was afterwards crushed and the coefficient of purity of the juice was determined. The results obtained by the refractometric analysis of these canes are put down in Table VII and plotted in Chart III. All the canes decidedly indicate, by the nature of the curves, immaturity to a greater or less extent. It will be seen from Table VIII that generally the ratio rises as the cane matures. But in no case is the ratio near unity. The reason for this is that owing to the late day in the season when the work was taken up, the canes had to be selected from a field which was

under harvest. In almost all the cases, the canes had to be cut from clumps carrying not more than 4 canes which were left as probably too young for crushing. It was, therefore, possible that all the canes were yet immature and constituted in each case a series of late shoots differing in age and maturity among themselves. It will also be seen that the coefficient of purity of the canes also generally varies as the top/bottom ratio, but no definite relationship seems to exist.

A comparison of the coefficients of purity obtained by us for each cane on the one hand with those cut for harvest from the same field in the same season amply supports the contention that the canes were all immature. In the same table are given both the sets of figures for the coefficients. It will be found that the coefficient of purity of the canes examined by us is always less than the corresponding bulk-harvest coefficients. There is therefore little reason to doubt their immaturity.

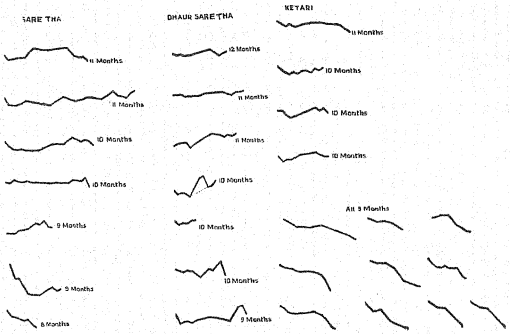
With a view to eliminate errors arising out of judging by the eye the stage of ripeness of the canes, experiments were arranged on an elaborate scale by the kind courtesy of the Government Sugarcane Expert. Four plots were reserved for this work exclusively at the cane-breeding Station and placed under our control and observation from the beginning of the season. As the shoots appeared they were labelled month after month as 1st, 2nd, 3rd and 4th shoots respectively according to their age. It was thought sufficient if each cane was marked as soon as cane formation set in above the ground level. The first shoots were labelled in July 1921, the second a month later in August, the third in September or October and the fourth in November.

Generally, according to our observations, the shooting is very liberal in the first month and this gradually decreases with the advance of time. Analysis of the canes thus marked was begun about the last week of December and were carried out at intervals of a month. The results presented in Table IX and Chart IV fully bear out the conclusions arrived at in 1919. As the canes become older and therefore more ripe, the curves also tend to become flatter and flatter.

In Table X will be found worked out the top/bottom ratios for the various canes against their age. In all these cases the age of the cane is definitely known and therefore the relative stages of maturity can be easily fixed. It will be found that there is a close relationship between the ratios and the stages of maturity. The former are characterized by their nearness to unity when the cane is mature with corresponding decreases at the younger stages. But what is more important than this confirmation of our results of the former

paper (1919) is the incidental proof that the ratio is independent of climatic and other conditions.

CHART IV.



Showing shapes of curves of labelled canes of different age.

It appears as though the effect of differences in climatic and soil conditions, while being widely different in the case of different canes, seems to be almost the same on the various parts of one and the same normal healthy cane, so far as the total-solid-contents are concerned. The work carried out in four different seasons shows a remarkable consistency in the general nature of the curves, considering the fact that we are dealing here with a living organism.

It is thus seen that from the nature of the curve formed by plotting the refractive indices of the juice of the internodes of any cane it is possible to judge of the stage of ripeness of that particular cane. As an alternativ rough and ready method the ratio of the total-solid-contents of the juices o. the bottom internodes and that of the internode carrying the highest dead leaf joint may also be used as a criterion in determining the ripeness of a cane. The latter method, however, cannot always be expected to give reliable results for the reason that the joint carrying the highest dead leaf attains to its maximum total-solid-content only some time after its leaf is cast or ceases to function. That this is the case has been shown in the 1919 paper already referred to. In a certain set of physiological experiments conducted for a different purpose we have noticed that it generally takes about three weeks

for a young internode to set its house in order and attain its optimum total-solid-content. If, therefore, the fixing of top by bottom ratio occurs before the internode is full, the ratio as determined from the highest dead leaf joint will naturally be low indicating immaturity, while in fact the cane is mature. The one striking feature of the curves of ripe canes is their relative flatness as distinguished from the definitely steep curves of the younger ones. This emboldened us to presume that generally if a cane is divided into two halves and crushed, the juices thus obtained contain practically the same amount of total solids in the case of a ripe cane. To put it in the language of the ratios, the ratio of the brix of the top juice to that of the bottom juice will be nearly unity ; and in the case of an unripe cane the ratio will be much less than unity. This method of arriving at " ratios " minimizes to a very great extent the uncertainties attendant on the fixing of ratios with single top and bottom internodes.

The next step in the course of the investigation is to see if this method could possibly be applied on the field scale. In Table XI are to be found the ratios thus obtained for four seasons at Palur. In all cases the analyses were conducted at a time when the agricultural officers in charge of the experiment station judged the crop to be ripe for harvest. Except in two cases the ratios are all on that side of 0·9 nearer to unity. It should be noted that these values were obtained not for canes cut up to the highest dead leaf joint, but for those cut in the way in which an ordinary ryot does for *jaggery* making i.e., a few internodes above the highest dead leaf joint.

Experiments were started during the last season this time also at the Sugarcane Breeding Station and by the kind permission of the Government Sugarcane Expert. The general scheme was to label sufficient number of canes early after planting and to analyse some of them periodically for the top and the bottom juices. This would enable us to compare the age of the cane with the ratios of top and bottom juices of canes of varying age. A few rows of Ketari, Saretha, B. Cheribon and Manjav were labelled for the first, second, third, and fourth shoots, etc. An interval of about a month and sometimes more was allowed between any two labellings. This would give us canes of varying age. Analysis was begun during the latter half of August and continued at intervals of a fortnight until the canes were dead ripe. The results are presented in Table XII. It will be found that as the cane advances in age, the ratio rises towards unity. But what is more to the point is that with such a rise in the ratio there is an increase in the percentage of total solids ; Ketari for which it was possible to get a complete set of results, fully supports the assumption. It is a native of Bihar, a vigorous growing variety,

which neither ripens early nor late. The ratio of the top and bottom juices rises towards unity from period to period of analysis and from one order of the shoot to an older one. This rise is further attended with definite increase in brix and sucrose content. Manjav shows a similar behaviour although it belongs to the class of thick or exotic canes. Black Cheribon, another thick variety, is slightly abnormal in its behaviour ; it was not in healthy condition and did not come off well in our experimental plot at the cane-breeding station.

Further experiments are in progress to test the application of this method to larger areas. A few trials of bulk analysis conducted on the above lines at Samalkot and Anakapalle are very encouraging. These and such others will form the subject of a separate communication.

Summary.

1. Work done during eight seasons show that, in general, as a cane ripens, the various internodes show a levelling tendency in the matter of their total-solid-contents.

2. The purity alone is not always a reliable criterion for judging the maturity of a cane crop.

3. In testing the stage of maturity of a cane, the ratio of top brix to bottom brix is quite handy and accurate.

We are indebted to Rao Sahib T. S. Venkatraman, Government Sugarcane Expert, and his assistants for the facilities afforded us in this investigation, particularly to Mr. Thomas who helped us a good deal in the preparation of labels, etc., and to Mr. Edmunds, Deputy Director of Agriculture, for allowing field scale tests on his crops at Samalkot and Anakapalle in 1922.

Note. Since submitting this paper for publication we have had access, about the middle of June 1924, to a new book by R. A. Quintus, entitled " The Cultivation of Sugarcane in Java," and through it the work of Dr. P. L. Lohr published in *Archief* 1920.

There is a close resemblance between the results obtained by Dr. Lohr and those obtained by us, and he comes to the same conclusion as ours in regard to the testing of the ripeness of sugarcane.

In discussing the determination of maturity of sugarcane, Quintus says : " Dr. P. L. Lohr proved that the tripartite analysis is sufficient and in some cases gives more reliable data than that of ten parts. The nearer the sugar percentages of the lower and upper parts approach each other the riper the cane is ; or rather, if these percentages approach each other the greatest possible quantity of available sugar is then present ..... " [B. V. N. and S. K.]

TABLE I.

Varying coefficients of purity in different soils and localities.

Name of variety	1918–19 Palur dry lands	Palur wet lands	Anakapalle	Samalkot	Limits between which the coefficient varies	1919–20 Gudiyatham	Palur dry lands	Anakapalle	Samalkot	Palur wet lands	Limits between which the coefficient varies
J 247 .. Bot. ..	65·59	76·97	79·24	91·28	58·38—91·28	91·14	83·40	80·9	78·70	92·65	78·70—90·55
J 247 .. Top ..	51·16 } 58·38					85·80 } 88·47	76·20 } 79·80			88·44 } 90·55	
B 208 .. Bot. ..	78·93	79·99	93·68	88·80	74·18—93·68	97·03	92·37	80·0	85·50	94·40	80·00—94·40
B 208 .. Top ..	69·42 } 74·18	75·62 } 77·81				90·45 } 93·74	84·85 } 88·61			94·41 } 94·40	
B 3412 .. Bot. ..	63·26	66·50	76·35	90·11	60·28—90·11	..	83·21	83·1	75·60	91·20	75·60—90·78
B 3412 .. Top ..	57·29 } 60·28						71·06 } 77·14			90·35 } 90·78	
B 1529 .. Bot. ..	78·42	78·43	93·56	91·78	77·44—93·56	96·45	91·88	70·1	87·40	94·86	70·10—96·17
B 1529 .. Top ..	76·46 } 77·44					93·86 } 96·17	91·56 } 91·72			93·57 } 94·22	
B 0450 .. Bot. ..	68·92	77·15	87·63	90·63	58·47—90·63	..	84·24	81·2	81·50	93·67	79·95—94·34
B 0450 .. Top ..	48·02 } 58·47						75·66 } 79·95			95·00 } 94·34	
Co 4	..	..	85·65	92·75	..	..	..	..	..	..	..
Co 7	..	..	89·00	84·09	..	..	..	..	..	..	..
Co 8	..	..	91·54	84·91	..	..	..	..	..	..	..
Co 12	..	..	82·99	92·33	..	..	..	..	..	..	..
Co 13	..	..	84·19	93·17	..	..	..	..	..	..	..

TABLE II.

Varying coefficients of purity in the same locality (Samalkot) for different seasons.

Name of variety	1917–18	1918–19	1919–20	1920–21	1921–22	Limits between which the values vary
Chinia ..	98·3	86·67	68·70	..	..	68·70—98·30
Chin	88·5	84·55	81·00	..	..	81·00—88·50
Barouka ..	93·5	76·69	67·70	..	..	67·70—93·50
Kharia ..	92·5	76·90	76·80	..	..	76·80—92·50
Pansahi ..	91·4	74·20	80·00	..	..	74·20—91·40
Saretha ..	99·2	81·83	80·70	..	..	80·70—99·20
Mungo ..	82·6	85·30	76·80	..	..	76·80—85·30
B 3412 ..	..	90·10	75·63	87·8	88·30	75·63—87·80
J 36 ..	..	91·30	84·90	90·7	82·40	82·40—91·30
Java Hebbal ..	..	94·20	89·10	93·0	86·30	86·30—94·20
CO. 1 ..	..	88·40	86·10	82·8	87·00	82·80—88·40

TABLE III.

Varying coefficients of purity in the same locality for different seasons.

Name of variety	1916–17		1917–18		1918–19		1919–20		1920–21	Limits between which the values vary
B 208										
Bot. ..	95·1	95·1	93·04	90·21	78·93	74·18	92·37	88·61	90·2	74·18—95·10
Top ..	95·1		87·38		69·42		84·85			
Fiji B										
Bot. ..	93·2	91·6	94·53	92·01	76·86	72·19	91·34	89·46	96·3	72·19—96·30
Top ..	90·0		89·49		67·51		87·59			
B 3412										
Bot. ..	91·8	90·5	89·51	85·72	63·26	60·28	83·21	77·14	85·90	60·28—90·50
Top ..	89·2		81·92		57·29		71·06			
J 247										
Bot. ..	90·3	90·5	89·15	88·48	65·59	58·38	83·40	79·80	91·7	58·38—91·70
Top ..	90·6		87·81		51·16		76·20			
B 1529										
Bot. ..	96·3	96·5	96·31	94·04	78·42	77·44	91·88	91·72	96·50	77·44—96·50
Top ..	96·7		91·77		76·46		91·56			
B 6450										
Bot. ..	91·3	90·9	92·88	91·44	68·92	58·47	84·24	79·95	90·5	58·47—91·44
Top ..	90·6		90·00		48·02		75·66			
Poovan										
Bot. ..	94·8	94·7	83·88	80·10	57·85	51·93	79·56	78·55	90·4	51·93—94·70
Top ..	94·6		76·32		46·00		77·54			
Vellai										
Bot. ..	91·0	91·8	94·12	92·74	62·06	55·74	87·22	85·34	90·5	55·74—92·74
Top ..	92·6		91·35		49·42		83·46			

TABLE IV.

Varying coefficients of purity in the same locality (Taliparamba) for different seasons.

Name of variety	1916-17	1917-18
B 6450	76·1	90·4
B 3412	82·4	90·0
Pansahi	79·9	88·4
Barouka	55·2	74·0
Saretha	85·0	81·8
Kari	78·7	84·8
Chinia	60·8	46·1

TABLE IV(*a*).

Variations of the purity coefficient with variations in the corresponding brix value.

Date of analysis	First shoots		Second shoots		Third shoots		Fifth shoots	
	Brix	Purity	Brix	Purity	Brix	Purity	Brix	Purity
21—VIII—1923 ..	17·57	84·86	17·07	85·24	16·27	82·42	15·16	79·68
10—IX—1923 ..	18·92	87·52	18·28	87·52	18·32	85·96	17·51	84·78
24—IX—1923 ..	19·05	88·14	18·70	88·78	18·44	88·08	18·19	86·76
12—X—1923 ..	19·40	89·40	19·35	89·00	19·25	88·50	18·14	89·40
Rise during interval	1·83	4·54	2·28	3·76	2·98	6·08	2·98	9·72
Rise of purity for 1 degree brix ..	..	2·50	..	1·65	..	2·00	..	3·20

TABLE V.

Showing the periodical rise in maturity coefficient (Extracted from the Formosa Agricultural Exp. Station Bull. No. 1).

Date	1912-13			1913-14		
	Sucrose	Reducing sugars	Maturity coefficient	Sucrose	Reducing sugars	Maturity coefficient
November 1 ..	5·44	1·303	4·20	10·70	1·587	6·7
,, 15 ..	9·35	1·071	8·70	9·50	1·623	5·9
,, 29 ..	8·12	0·832	9·80	10·77	1·072	10·0
December 26 ..	13·38	0·687	19·50	12·40	0·804	15·4
January 9 ..	13·58	0·198	70·10	13·49	0·188	71·8
February 20 ..	13·04	0·163	80·00	14·51	0·772	18·8
March 6 ..	13·10	0·051	256·79	13·91	0·174	79·9

TABLE VI.

Showing rise in total solids of the juices of a few internodes of canes during intervals of a month.

Name of cane	Number of internode from bottom	Analysed in Dec.	Rise during Dec.-Jan.	Analysed in Jan.	Rise during Jan.-Feb.	Analysed in Feb.	Rise during Feb.-Mar.	Analysed in Mar.
B 208 ..	2	22·42	0·89	23·31	−0·14	23·17	−0·04	23·13
	5	21·70	1·05	22·75	0·46	23·21	−0·04	23·17
	8	18·35	4·06	22·41	1·31	23·72	−0·92	22·82
	12	..	..	21·58	2·41	23·99	−0·78	23·21
Yerra ..	2	20·33	2·24	22·57	0·18	22·75	−1·73	21·02
	5	19·09	3·48	22·57	0·75	23·32	−0·92	22·42
	8	18·09	3·90	21·99	0·95	22·94	0·67	23·62
	10	..	..	21·39	0·74	22·13	0·23	22·36
Mogali ..	2	19·80	1·89	21·69	−0·89	20·80	−0·37	20·43
	6	18·12	3·00	21·12	0·89	21·99	−0·06	21·93
	9	16·27	4·25	20·52	1·64	22·26	−0·02	22·24
	13	..	..	20·52	2·69	23·21	−0·43	22·78
Majorah ..	2	17·97	0·58	18·55	0·07	18·62	0·01	18·63
	5	18·18	0·10	18·28	0·65	18·93	0·30	18·63
	8	15·84	1·84	17·71	1·52	19·23	−0·89	18·34
	12	14·62	3·13	17·75	1·74	19·49	−1·72	17·77
	17	12·12	4·98	17·10	2·74	19·84	−0·87	18·97
Kharia ..	2	17·02	1·59	18·61	0·02	18·63	0·45	19·08
	5	16·75	3·10	19·85	−0·07	19·78	0·25	20·03
	8	16·13	4·92	21·05	−0·62	20·43	0·12	20·31
	11	14·58	5·27	19·85	1·48	21·33	−1·58	19·75
Kaludai Boothan	1	19·28	0·43	19·71	0·42	20·13	1·96	22·09
	5	18·07	1·91	19·98	1·09	21·07	1·31	22·38
	10	16·50	3·42	19·92	1·97	21·89	0·59	24·47
Cheni ..	1	16·02	1·53	17·55	1·68	19·23	..	..
	5	15·42	1·78	17·20	1·88	19·08	..	..
	10	12·10	6·85	18·95	0·35	19·30	..	..

TABLE VII.

Total solid contents (calculated as sucrose) of various internodes of a cane as determined by the Refractometer during April 1921.

Number of the internode from the base	Name of variety with the probable stage of maturity													
	D 1135			H 109		B 1528			D 74			Sanachi		
	Early	Inter.	Late	Early	Late	Early	Late	Inter.	Late	Early	Shoot	Early	Inter.	Late
1	22·73	23·74	22·47	23·35	22·12	23·32	24·50	24·30	20·68	23·72	22·80	20·17	21·23	19·24
2	22·67	24·14	21·45	23·35	22·05	23·02	23·10	23·70	20·13	22·86	22·57	19·12	19·18	19·59
3	21·82	22·74	22·85	23·07	21·45	22·87	20·72	22·80	19·33	22·51	22·87	18·92	18·73	17·24
4	21·02	21·73	21·95	22·82	21·20	21·51	18·67	21·87	18·63	22·41	23·02	18·52	17·33	17·18
5	21·02	22·18	18·45	22·32	19·75	21·26	15·82	21·42	18·28	21·56	22·27	18·42	17·68	17·18
6	20·27	21·98	17·85	21·23	19·20	21·46	16·17	19·97	17·73	21·41	22·02	18·42	17·03	17·18
7	21·01	21·85	17·85	21·19	19·25	20·66	14·07	18·07	17·53	21·46	21·92	18·37	16·93	16·58
8	21·01	20·55	18·60	20·94	18·05	20·66	..	17·52	18·28	20·21	20·94	18·07	17·03	16·08
9	21·16	20·55	18·18	20·24	18·20	19·98	..	17·82	17·93	19·81	20·49	18·17	17·03	15·43
10	21·01	20·35	17·78	19·32	..	19·98	..	17·89	18·03	19·81	18·74	18·17	16·78	15·93
11	20·59	20·02	16·87	19·32	..	19·33	..	16·39	17·38	19·56	18·19	18·42	16·68	15·82
12	20·44	20·52	16·87	19·67	..	19·08	..	17·34	17·38	20·21	19·74	17·92	15·93	15·52
13	19·59	20·92	16·57	19·47	..	19·68	..	17·34	16·38	19·01	17·39	17·92	15·48	14·86
14	18·99	20·62	16·27	19·67	..	19·33	..	..	..	18·11	12·79	17·82	16·02	13·66
15	19·39	19·67	16·17	18·42	..	18·13	..	15·79	..	18·81	..	17·27	16·33	13·21
16	18·58	19·17	15·67	17·97	..	17·88	..	..	..	17·46	..	17·77	16·53	13·21
17	18·58	19·12	..	17·82	..	16·28	..	..	..	18·41	..	18·42	15·78	12·91
18	16·83	18·88	..	..	..	16·28	..	..	..	19·61	..	17·87	14·78	12·91
19	16·17	18·48	..	..	..	16·38	..	..	..	19·21	..	18·42	13·53	11·86
20	16·71	17·23	..	..	..	..	..	..	..	18·71	..	17·47	13·53	..
21	..	17·23	..	..	..	..	..	..	..	19·91	..	17·82	13·12	..
22	..	17·23	..	..	..	..	..	..	..	18·36	..	17·82	..	..
23	..	..	..	..	..	..	..	..	..	17·16	..	18·22	..	..
24	..	..	..	..	..	..	..	..	..	16·11	..	16·32	..	..
25	..	..	..	..	..	..	..	..	..	..	..	16·17	..	..
26	..	..	..	..	..	..	..	..	..	..	..	16·17	..	..
27	..	..	..	..	..	..	..	..	..	..	..	16·17	..	..

TABLE VIII.

$\frac{Top}{Bot.}$ ratios of canes analysed in 1921 *with their purities and the purities obtained by bulk-crushing of the same variety in the same season, place and plot.*

Name of variety	EARLY		INTER.		LATE		Coefficient of purity * (standard)
	$\frac{Top}{Bottom}$	Coefficient of purity	$\frac{Top}{Bottom}$	Coefficient of purity	$\frac{Top}{Bottom}$	Coefficient of purity	
B 3412 ..	0·85	85·74	..	..	..	..	84·0
D 1135 ..	0·74	85·45	0·73	85·49	0·70	80·10	91·0
H 109 ..	0·76	82·90	..	..	0·82	80·90	88·7
B 1528 ..	0·70	86·96	0·65	83·99	0·57	69·10	90·0
Sanachi ..	0·80	83·73	0·62	74·33	0·60 0·54	70·60	..
D 74 ..	0·68	..	..	..	0·60 0·56	81·51 79·97	85·7

* The figures under this column are the purities of the juices of corresponding varieties cut from the same field in the same year for harvest.

TABLE IX.

Showing total solid contents (calculated as sucrose), as determined by the Refractometer, of the various internodes of canes labelled and analysed during 1921–1922.

Saretha.

No. of internode from the base	Order of shoot	First		Second			Third	Fourth	
	Labelled on—	JULY 1921		AUGUST 1921			OCTOBER 1921	NOVEMBER 1921	
	Analysed on—	16-XII-1921	26-I-1922	25-XII 1921	27-I-1922	28-II-1922	6-III-1922	28-II-1922	6-III-1922
1 ..	..	15·08	18·45	13·49	18·45	15·47	..	10·41	17·63
2 ..	..	14·78	17·10	13·49	17·60	14·57	20·36	9·26	17·03
3 ..	..	15·53	17·05	13·49	17·10	14·72	18·06	8·91	16·38 { Borer
4 ..	..	15·23	17·05	14·01	16·90	14·87	18·06	8·64	16·38 { attack.
5 ..	..	15·13	17·40	..	17·10	..	..	8·29	17·03
6 ..	..	15·23	17·85	14·44	16·90	14·94	15·32	8·29	..
7 ..	..	15·13	..	..	..	..	15·10	7·64	..

TABLE IX—*contd.*

Saretha—*contd.*

No. of internode from the base	Order of shoot	First		Second			Third	Fourth	
	Labelled on—	JULY 1921		AUGUST 1921			OCTOBER 1921	NOVEMBER 1921	
	Analysed on—	16-XII-1921	26-I-1922	25-XII-1921	27-I-1922	28-II-1922	6-III-1922	28-II-1922	6-III-1922
8	..	..	..	15·39	..	16·79	..	6·94	..
9	..	..	17·50	15·19	16·75	..	15·10	..	..
10	..	..	..	15·79	..	..	..	..	..
11	..	14·78	..	14·69	..	16·44	16·23	..	..
12	..	..	17·10	14·59	17·85	..	16·48	..	..
13	..	14·88	..	..	..	..	15·88	..	..
14	..	..	..	..	..	16·74	16·23	..	..
15	..	..	18·60	..	17·85	..	..	..	..
16	..	14·88	..	..	18·45	16·79	..	..	..
17	..	15·40	..	..	19·10	..	..	..	..
18	..	15·30	17·70	..	16·30	15·21	..	..	..
19	..	15·20	..	..	18·45	15·01	..	..	..
20	..	15·70	..	..	18·60	15·11	..	..	..
21	..	14·00	18·45	..	18·35	14·31	..	..	..
22	..	..	..	..	17·50	..	..	..	..
23	..	..	..	..	..	..	..	..	..
24	..	..	18·10	..	..	..	..	..	..
25	..	..	..	..	..	..	..	..	..
26	..	..	..	..	..	..	..	..	..
27	..	..	19·50	..	..	..	..	..	..
28	..	..	18·60	..	..	..	..	..	..
29	..	..	18·45	..	..	..	..	..	..
30	..	..	18·30	..	..	..	..	..	..
31	..	..	19·05	..	..	..	..	..	..
32	..	..	19·50	..	..	..	..	..	..

TABLE IX.—*contd.*

Dhaur Saretha.

No. of internode from the base	Labelled on—	First shoot				Second shoot			
		JULY 1921				AUGUST 1921			
	Analysed on—	26-XII-1921	28-I-1922	1-II-1922	5-III-1922	25-XII-1921	28-I-1922	30-I-1922	5-III-1922
1 ..	..	20·46	21·38	21·70	22·34	16·19	22·50	16·10	21·72
2 ..	..	17·78	21·73	21·80	22·09	14·51	21·95	15·35	21·77
3 ..	..	20·01	..	21·80	22·09	14·84	22·10	15·50	21·52
4 ..	..	20·23	21·88	21·45	21·94	14·86	22·10	15·35	21·52
5 ..	..	20·23	21·08	21·45	..	14·71	22·50	14·90	20·32
6 ..	..	..	21·70	..	22·24	14·96	22·50	..	20·92
7 ..	..	19·03	..	21·45	..	..	..	18·45	22·42
8 ..	..	..	22·65	..	22·64	..	21·40	18·80	22·12
9 ..	..	20·53	..	21·95	..	15·53	..	16·65	..
10 ..	..	20·33	23·45	..	23·03	..	21·65	16·90	..
11 ..	..	..	..	22·05	..	15·33	..	17·85	..
12 ..	..	21·08	23·20	..	22·08	..	21·65	..	..
13 ..	..	21·83	22·95	22·05	22·38	..	..	..	..
14 ..	..	19·33	23·20	..	22·63	14·73	..	..	..
15 ..	..	..	23·10	21·45	..	16·18	..	..	..
16 ..	..	..	23·35	21·95	..	17·38	..	..	..
17 ..	..	..	..	22·05	..	17·58	..	..	..
18 ..	..	..	..	22·05	..	16·23	..	..	..

TABLE IX.—*concld.*

Ketari.

No. of internode from the base	Order of shoot—	First				Second		Third	Fourth						
	Labelled on—	AUGUST 1921				SEPTEMBER 1921		OCTOBER 1921	NOVEMBER 1921						
	Analysed on—	26-XII-1921	24-I-1922	1-II-1922	23-II-1922	1-II-1922	23-II-1922	27-II-1922	16-III-1922	16-III-1922	20-III-1922	20-III-1922	20-III-1922	22-III-1922	23-III-1922
1	..	19·53	18·95	17·40	18·08	18·85	16·59	18·19	..	..	15·96	16·64	..	..	16·03
2	..	18·98	18·20	17·25	17·48	17·90	15·94	17·59	15·46	18·56	..	..	15·81	14·58	..
3	..	18·28	18·35	16·70	17·12	17·25	15·34	17·39	..	..	14·21	15·09	..	..	15·88
4	..	18·03	18·45	16·05	17·37	17·15	15·14	17·24	14·86	..	..	..	15·16	13·93	..
5	..	18·03	..	..	16·97	17·50	15·44	..	..	17·31	14·51	14·94	..	..	15·58
6	..	..	19·25	16·70	..	17·00	15·44	16·94	14·86	..	..	..	15·16	13·86	..
7	..	17·98	..	..	17·27	17·00	15·74	..	..	..	12·41	15·39	..	..	13·86
8	..	..	19·45	17·30	..	17·00	15·83	16·89	13·86	17·01	..	..	14·21	13·31	..
9	..	18·23	..	..	17·57	15·45	15·48	..	..	..	11·61	14·44	..	..	12·21
10	..	..	19·60	17·90	..	14·85	16·23	16·39	12·66	..	..	13·59	11·91	12·21	..
11	..	17·58	19·25	17·40	17·57	..	15·83	15·99	12·51	17·46	10·91	..	..	11·96	..
12	..	16·83	18·85	17·65	..	..	16·23	15·04	..	..	..	..	11·26	..	..
13	..	15·66	18·85	17·00	17·39	..	..	13·34	..	..	..	..	..	..	..
14	..	15·06	..	..	..	..	..	..	..	16·51	..	..	10·61	..	..
15	..	..	..	..	17·24	..	..	..	..	..	..	..	..	..	..
16	..	..	..	..	17·14	..	..	..	..	..	..	..	..	..	..
17	..	..	..	..	..	..	..	..	..	15·46	..	..	..	..	..
18	..	..	..	..	15·99	..	..	..	..	..	..	..	..	..	..
19	..	..	..	..	..	..	..	..	..	14·66	..	..	..	..	..

TABLE X.

Ratio of $\frac{\text{Total solids of the top internode}}{\text{Total solids of the bot. internode}}$ *of canes with their age.*

Name of variety	8 months old	9 months old	10 months old	11 months old	12 months old
Saretha	0·67	1·08	0·94	0·93	....
	....	....	0·94	1·06	....
Dhaur Saretha ..	....	1·00	0·95	1·02	1·01
	....	....	1·00	1·02	....
	....	....	1·11	1·09	....
Ketari	....	0·74	0·99	....	....
	....	0·79	0·98	....	....
	....	0·77	0·98	....	....
	....	0·68	....	....	....
	....	0·81	....	....	....
	....	0·82	....	....	....
	....	0·87	....	....	....

TABLE XI.

Ratio of $\frac{\text{Brix of Juice of Top half}}{\text{Brix of Juice of Bot. half}}$ *of canes examined.*

Name of variety	1916-17	1917-18	1919-20	1921-22
Fiji B	0·99	0·93	0·99	0·87
B 208	0·97	0·94	0·94	0·97
B 1529	0·97	0·89	0·93	0·92
B 147	0·97	....	0·90	0·86
Vellai	1·00	0·98	0·93	0·94
B 6450	0·95	0·91	0·98	0·92
B 3412	0·97	0·89	0·96	0·95
J 247	0·98	0·93	0·89	0·89
Green Sports	1·00	0·94	0·94	....
Poovan	0·95	1·09	1·03	....
Red Mauritius	0·95	0·94	0·94	....
Striped Mauritius	1·03	0·97	1·03	0·94
Ashy Mauritius	0·98	0·96	0·99	0·84
Fiji C	0·96	0·94	0·92	....

TABLE XII.

Showing rise of the ratio of $\frac{\text{Top half}}{\text{Bot. half}}$ *towards unity with advancing age along with the brix, sucrose and purity of the juices analysed.*

Name of variety	Analysed on—	Order of shoot	Brix	Sucrose	Purity	Brix		Ratio
						Bot.	Top	$\frac{\text{Top}}{\text{Bot.}}$
Ketari ..	21-VIII-1923	First	17·57	14·81	84·86	18·62	16·50	0·89
		Second	17·07	14·55	85·24	18·72	15·20	0·81
		Third	16·27	13·41	82·42	18·01	14·70	0·81
		Fifth	15·16	12·07	79·68	17·11	13·49	0·79
	10-IX-1923	First	18·92	16·56	87·52	19·52	18·40	0·95
		Second	18·28	15·91	87·52	19·22	17·41	0·91
		Third	18·32	15·75	85·96	18·62	17·74	0·93
		Fourth	..	..	..	..	..	..
		Fifth	17·51	14·84	84·98	18·77	16·27	0·87
	24-IX-1923	First	19·05	16·79	88·14	19·52	18·41	0·94
		Second	18·70	16·60	88·78	19·52	17·81	0·91
		Third	18·44	16·24	88·08	19·12	17·40	0·91
		Fourth	..	..	..	..	..	..
		Fifth	18·19	15·78	86·76	18·92	17·00	0·90
	12-X-1923	First	19·40	17·28	89·06	19·37	19·17	0·99
		Second	19·35	17·22	88·98	19·48	19·12	0·98
		Third	19·25	17·12	88·93	19·48	18·72	0·96
		Fourth	18·78	16·78	89·35	18·60	17·70	0·95
		Fifth	18·14	16·22	89·41	18·57	17·40	0·94
	3-XI-1923	First	19·32	17·33	88·00	19·32	19·42	1·01
		Second	18·32	16·36	89·30	18·22	18·22	1·00
Manjav ..	22-VIII-1923	First	18·45	16·45	89·16	19·52	17·30	0·89
		Second	15·74	13·29	84·43	18·32	13·39	0·72
		Fifth	14·12	11·09	78·54	17·10	11·08	0·65
	11-IX-1923	First	18·44	16·37	88·78	19·83	16·93	0·85
		Second	16·84	14·60	86·70	19·03	14·57	0·77
		Third	18·34	16·08	87·26	18·98	17·87	0·94
		Fifth	15·54	12·77	82·19	18·18	13·30	0·73
	24-IX-1923	First	18·67	16·87	90·36	19·31	18·11	0·94
		Third	18·30	16·41	89·67	19·31	17·20	0·89
B-Cheribon .	22-VIII-1923	First	17·34	15·19	87·80	19·12	15·50	0·81
		Second	17·04	14·87	87·41	19·62	14·10	0·72
		Third	15·34	13·24	86·30	15·60	15·00	0·96
		Fourth	17·54	15·69	89·60	19·07	15·90	0·83
		Fifth	16·84	14·79	87·80	18·41	15·20	0·83
	11-IX-1923	First	17·87	15·82	88·55	18·59	16·98	0·91
		Second	17·81	15·61	87·66	19·19	16·22	0·85
		Fifth	17·97	15·76	87·70	19·07	16·62	0·87

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TABLE XII.

Showing rise of the ratio of $\frac{Top\ half}{Bot.\ half}$ towards unity with advancing age along with the brix, sucrose and purity of the juices analysed.

Name of variety	Analysed on—	Order of shoot	Brix	Sucrose	Purity	Brix		Ratio
						Bot.	Top	$\frac{Top}{Bot.}$
Kotari ..	21-VIII-1923	First	17·57	14·81	84·86	18·62	16·50	0·89
		Second	17·07	14·55	85·24	18·72	15·20	0·81
		Third	16·27	13·41	82·42	18·01	14·70	0·81
		Fifth	15·16	12·07	79·68	17·11	13·49	0·79
	10-IX-1923	First	18·92	16·56	87·52	19·52	18·40	0·95
		Second	18·28	15·91	87·52	19·22	17·41	0·91
		Third	18·32	15·75	85·96	18·62	17·74	0·93
		Fourth	..	..	..	..	..	..
		Fifth	17·51	14·84	84·98	18·77	16·27	0·87
	24-IX-1923	First	19·05	16·79	88·14	19·52	18·41	0·94
		Second	18·70	16·60	88·78	19·52	17·81	0·91
		Third	18·44	16·24	88·08	19·12	17·40	0·91
		Fourth	..	..	..	..	..	..
		Fifth	18·19	15·78	86·76	18·92	17·00	0·90
	12-X-1923	First	19·40	17·28	89·06	19·37	19·17	0·99
		Second	19·35	17·22	88·98	19·48	19·12	0·98
		Third	19·25	17·12	88·93	19·48	18·72	0·96
		Fourth	18·78	16·78	89·35	18·60	17·70	0·95
		Fifth	18·14	16·22	89·41	18·57	17·40	0·94
	3-XI-1923	First	19·32	17·33	88·00	19·32	19·42	1·01
		Second	18·32	16·36	89·30	18·22	18·22	1·00
Manjav ..	22-VIII-1923	First	18·45	16·45	89·16	19·52	17·30	0·89
		Second	15·74	13·29	84·43	18·32	13·39	0·72
		Fifth	14·12	11·09	78·54	17·10	11·08	0·65
	11-IX-1923	First	18·44	16·37	88·78	19·83	16·93	0·85
		Second	16·84	14·60	86·70	19·03	14·57	0·77
		Third	18·34	16·08	87·26	18·98	17·87	0·94
		Fifth	15·54	12·77	82·19	18·18	13·30	0·73
	24-IX-1923	First	18·67	16·87	90·36	19·31	18·11	0·94
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		Third	15·34	13·24	86·30	15·60	15·00	0·96
		Fourth	17·54	15·69	89·60	19·07	15·90	0·83
		Fifth	16·84	14·79	87·80	18·41	15·20	0·83
	11-IX-1923	First	17·87	15·82	88·55	18·59	16·98	0·91
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BY

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AGRICULTURAL RESEARCH INSTITUTE, PUSA

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PREFACE

THE only phosphatic deposit of importance in Southern India consists of the Septarian nodules of phosphate of lime, found in the cretaceous beds of Trichinopoly and estimated at 8-million tons thirty-two years ago by Dr. Warth. Miners and manure firms sought in earlier years to profit themselves by exporting the material to other countries, and would have completed its removal by this time if it were less impure and more suitable for the manufacture of superphosphate. Little or no attempts were made to utilize it for fertilizing the soils of South India which are particularly deficient in phosphoric acid.

Investigation on the utilization of the powdered phosphatic nodules for paddy, the most important food crop of the Madras Presidency, was begun by the author as early as 1914, but he could not devote undivided attention to it, as any research work could be undertaken by him only so long as it did not interfere with his main duties at the College, namely, teaching, which, however, absorbed a considerable portion of his time all along. Dr. W. H. Harrison, who was Government Agricultural Chemist and under whom the author had the pleasure of working as his chief assistant at the time, provided him with facilities to tour several times in the area of phosphatic nodules, to consult records pertaining to the area in the Collector's office, Trichinopoly, and to carry on experiments on the growth of paddy in pots in the pot culture house at Coimbatore.

Isolated portions of the subject of this memoir were dealt with by the author, now and then, during these 10 years. He read papers on the "Economic possibilities of the Cretaceous Formation in Southern India" and "The availability of flour phosphate as a phosphatic manure for paddy," illustrated with lantern slides, before the Geology and Agricultural Sections of the Indian Science Congress held in 1915, 1920 and 1924. He read a paper, illustrated with charts and diagrams, on "The phosphatic problem in Southern India" at the Agricultural Conference held at Coimbatore in 1917, an abstract of which was published in the *Journal* of the Union. A preliminary account, chiefly descriptive, of the "Phosphatic nodules of Trichinopoly" was published in the *Year Book* of 1918 of the Madras Agricultural Department; and this publication attracted the attention of Government, manure firms and the

public, with the result that the Madras Agricultural and Industrial Departments seriously considered working the area as a departmental concern, and manure firms and private parties took out mining leases of some of the blocks within the last few years. The present memoir details the complete work of the author on the phosphatic nodules of Trichinopoly on which the author has been engaged from 1914 up to date.

The object of the research was to ascertain how far the powdered phosphatic nodules can be used as a manure for supplying phosphoric acid to the paddy plant. It does not aim at solving the question whether it would pay economically. Being an insoluble substance and not capable of being washed out of the soil, applications even in small quantities repeated year after year will add to the phosphorus content of the soil, and one may say without hesitation, whether it will pay or not in the year of application, that it will pay in the long run when applied, along with organic matter, to soils deficient in phosphoric acid, as most deltaic soils in the Presidency are.

The author is indebted to several gentlemen who helped him in this work, and his thanks are gratefully rendered to them : to Dr. W. H. Harrison for help and advice, to Dr. R. V. Norris for suggestions, to his assistants, Messrs. C. V. Ramaswami Ayyar, H. Shiva Rao and N. Krishna Ayyar, for the analyses of some of the composts, to the Deputy Directors of Agriculture, IV, V and VIII Circles, and their subordinate officers and the gentlemen in these Circles who placed their lands at his disposal, for carrying on co-operative experiments, and to Mr. T. Lakshmana Rao and the College Museum Curator for the photographs.

The Agricultural College and Research Institute,
Coimbatore, Madras,
6th June, 1924.

M. R. RAMASWAMI SIVAN.

CONTENTS

THE PHOSPHATIC NODULES OF TRICHINOPOLY AND THE AVAILABILITY OF FLOUR PHOSPHATE AS A MANURE FOR PADDY.

BY

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PART I.

HISTORICAL AND DESCRIPTIVE.

OF all the geological formations in Peninsular India, probably none are more interesting than the cretaceous beds of Trichinopoly, containing well preserved fossils in the midst of azoic rocks of by-gone ages. Apart from a few isolated earlier observers, a party of the Geological Survey surveyed the locality over half a century ago. The marine fossils were, however, more interesting to Blanford,[1] who was in charge of the party, than the septarian nodules about which he makes nothing more than a passing reference and which, he surmises, "may have been formed round similar organisms, the impression of which has disappeared in the subsequent shrinking of the interior and the infiltration of calcspar and gypsum which fill the septaria."

In January 1892, 32 years later, Dr. Warth, Superintendent of the Madras Central Museum, visited the locality to collect fossils for the museum, and he reported to Government "the existence of a large area in Perambalur Taluk, strewn over with nodules of phosphate of lime lying on the surface," and reported later that the deposit was of real economic importance, "the quantity being great, reckoned by the square miles of the tract where the deposits were found", and that "the nodules just picked off from the surface would alone amount to 4,000 tons."

[1] *Mem. Geo. Sur. of Ind.*, 1862, Vol. IV, Pt. I, p. 83.

In view of the increasing demand for the nodules from different manure firms, Dr. Warth was directed by Government to conduct a more detailed inspection of the locality and submit a proper estimate of the nodules, in pursuance of which, he made an examination of the ground along a measured line of 6,400 feet across the tract from Uttatur and confirmed his previous estimate of surface deposits. He also made a number of excavations to different depths and, from a study of the strike and dip of the strata, concluded

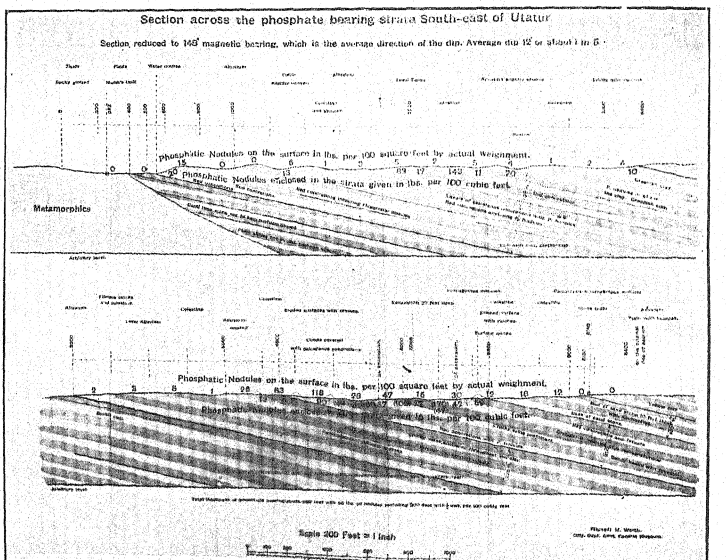


FIG. 1. Section across phosphate bearing strata.

that the nodule-bearing strata were at least 1,150 feet thick, that 100 cubic feet of clay would yield on an average 28 lb. of nodules, and that from 7 to 8 million tons of nodules were present to a depth of 200 feet. The photograph of the map prepared by Dr. Warth is reproduced here[1] (Fig. 1). Government immediately ordered the locality to be surveyed by the Revenue Survey

[1] G. O. No. 821, Rev., dated 14th September, 1893 contains the final report of Dr. Warth.

Department, who demarcated the same into 9 blocks of over one square mile each[1] (Fig. 3).

An account of the phosphatic nodules is given in *Agricultural Ledger* No. 20 of 1898 by D. Hooper, Government Quinologist, extracted partly from the old Memoirs of the Geological Survey and partly from Dr. Warth's reports; and a preliminary account, chiefly descriptive, of the phosphatic nodule area was published by the author in the *Year Book* of the Madras Agricultural Department early in 1918; and, from this period, manure firms, co-operative societies, private parties and the Government of Madras directed their serious attention to the subject of the utilization of the phosphate.

A short description of the locality and the manner in which the nodules are distributed will be given below :—

The principal village of Uttatur (Fig. 2) in the locality is connected by a three-mile gravelled road with the Trichinopoly-Madras Trunk Road, which

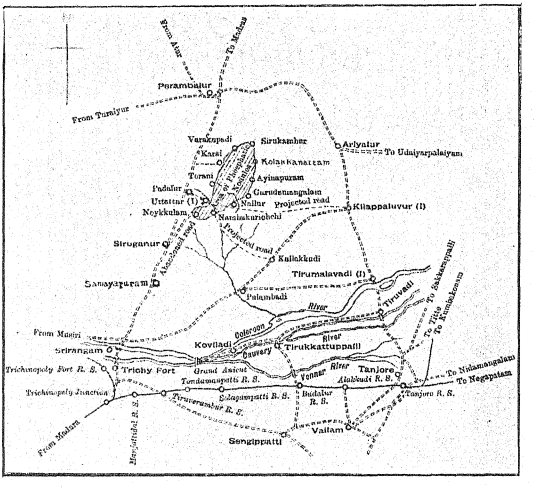


FIG. 2. Map of portions of Trichinopoly and Tanjore Districts showing the positions of the area of phosphatic nodules.

[1] Mining Block Maps, I to IX, are available at the Collector's office, Trichinopoly, at one rupee each, drawn to a scale of 1 inch to a mile and showing survey numbers included in each mining block, Inam, Poramboke or Government area and nature of cultivation, if any.

it joins at the 21st mile from Trichinopoly Fort. A new road was proposed to be constructed from Uttatur to Kilappaluvur, whereby its distance to Tanjore would be about 30 miles across the Coleroon. In other words, the locality is within easy distance of the Tanjore paddy tracts. When the long projected Villupuram-Trichinopoly Railway becomes an accomplished fact—the line is now being surveyed—the line will pass close to the locality.

The locality of phosphatic nodules is situated in a long strip of land, over 10 miles long from Sirugambur on the north to Neikkulam on the south and approximately one to two miles in breadth and consists of portions of the villages of Terani, Teranipalayam, Karai, Varagampadi, Sirugambur, Kolakanatham, Ainapuram, Garudamangalam, Nallur, Nambikurichi, Neikkulam and the inam devastanam village of Uttatur (Fig. 3).

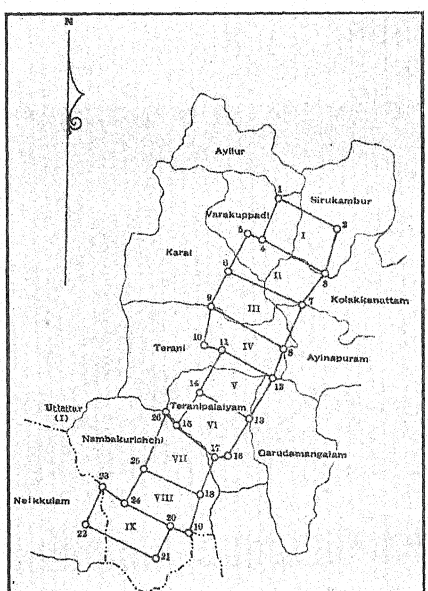


FIG. 3. Locality of phosphatic nodules, Trichinopoly District.

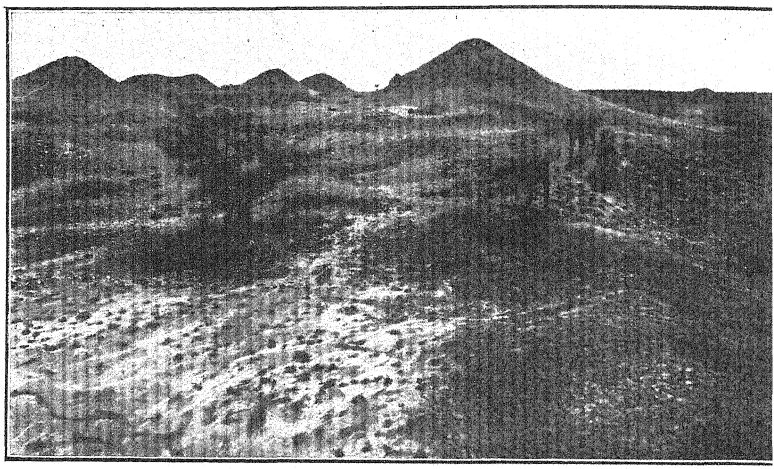


FIG. 1. General view of mounds.

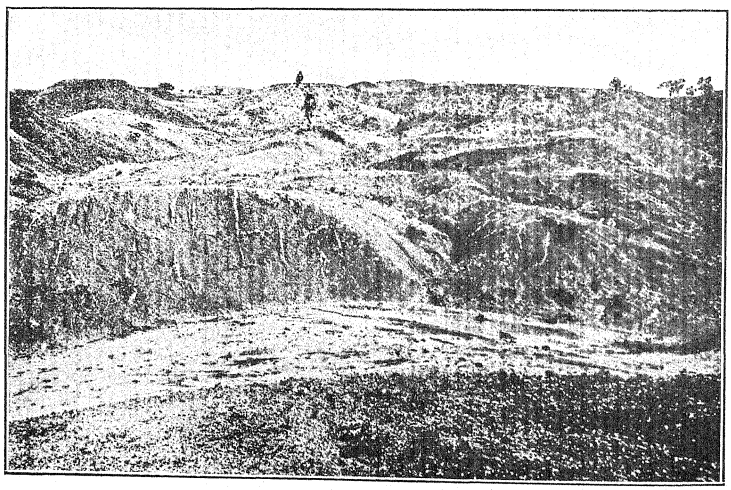


FIG. 2. Mounds showing white lines on denuded slopes.

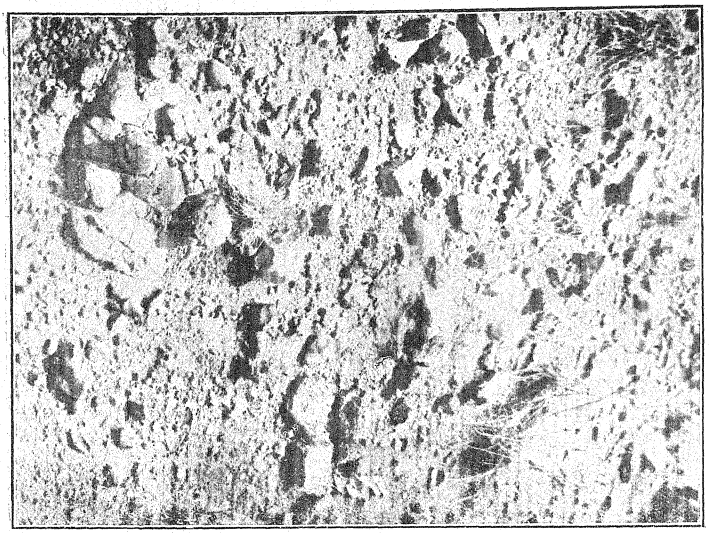


FIG. 1. Nodules exposed *in situ*.

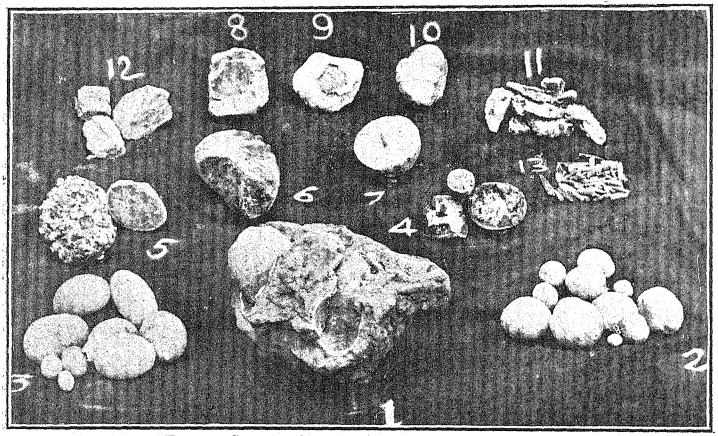


FIG. 2. Some geological specimens of the locality.

(1) Phosphatic nodules, imbedded in yellow clay, *in situ*. The nodules may be spherical (2) or oval (3). The phosphatic nodules, when broken, give a peculiar crystalline fracure (4), which is absent in purely calcareous nodules (7). When the phosphatic nodules undergo weathering the dark core inside is seen (5). Sometimes the phosphates may be present in other forms than the septaria (6). The nodules are enclosed in a rind of hardened shale (10), sometimes chalk (9). Between the rind and the nodules, there is usually a layer of crystalline gypsum (8). The region of phosphatic nodules abounds in gypsum (11), celestine (12) and belemnites (13).

On the west are detached hills of azoic rocks, on the north is broken open country, on the east are the younger cretaceous formations overlying the older Uttaturs, and on the south the drainage is carried off by a number of ravines into the Nandyar river which flows into the Coleroon. The enclosed tract is made up of yellow shales, which are occasionally white, cut up, through the denudation of centuries by several gullies, into a number of small mounds on the tops of which well polished ferruginous concretions of rich hæmatite are found strewn on the surface (Plate I, fig. 1). In these conical heaps of soft yellow clay are seen white lines of chalk, two to three inches in diameter, branching in all directions, on the surface and underground, but better exposed on denuded slopes of ravines (Plate I, fig. 2). On these lines, round or oval stones are attached, usually enclosed, partly or wholly, in a rind of hardened shale or limestone and often separated by a layer of crystalline gypsum. These stones are the phosphatic nodules. They are said, by the villagers, to grow up after heavy rains; in other words, when the soft yellow clay is washed out by rain, the nodules underneath are exposed and found strewn on the surface (Plate II, fig. 1). They are of all sizes, from one inch to ten inches in length and from half inch to five inches in breadth, usually oval and sometimes spherical. Occasionally the phosphate of lime is found in other forms than the septaria. They show a peculiar crystalline structure when broken, which distinguishes them from calcareous nodules. The nodules, when subjected to weathering action, lose their white covering and expose the dark core inside. Crystalline gypsum in flakes is abundant and characteristic of the locality; and celestine, chalk and belemnites are also found associated with the phosphatic nodules (Plate II, fig. 2). The nodules are present only in the region of yellow clay and are completely absent in the black soils of the immediate vicinity. The nodules on the surface have been mostly removed from Uttatur, Nambikurichi, Teranipalayam and Karai by petty contractors who, in earlier years, delivered the nodules at Rs. 12 per ton at Trichinopoly Fort where a few agents bought and supplied the same to the manure firms. There was a lot of correspondence and much controversy between the Government of Madras and the manure firms regarding the terms of mining lease and between the Departments of Agriculture and Industries as regards the advisability of working the deposit as a Government concern, and reference may be made to the article in the *Year Book* of 1918 of the Madras Agricultural Department published by the author.

It is a matter for regret that, in an agricultural country like India, a large export trade in bones should exist, amounting to 107,843 tons for nine

months of the year 1920, valued at £1,102,051[1] and that the few phosphatic deposits of any value should not be worked. The only sources in the Presidency other than the phosphatic nodules of Trichinopoly seem to be the small quantities of apatite in the waste of the Nellore mica mines and the isolated crystalline forms of mangan-apatite in the Kodurite series of the manganese mines of Vizagapatam; and all that can be said about these forms of apatite is that very little is known about them and the quantity seems to be negligible.*

PART II.

EXPERIMENTAL.

1. Composition of flour phosphate.

The analyses of South Indian soils, especially the soils of the Cauvery delta in Tanjore,[2] show a general deficiency in phosphoric acid. As many of these soils are double crops wet land and as the grain is largely exported from the tract, the depletion of the phosphatic ingredient in these soils continually goes on, though partly recouped by river silt, and the necessity of utilizing all the phosphatic manure in the country distinctly arises. Besides bonemeal and fish manure—which, by-the-bye, are largely exported—the nodules of Trichinopoly are the most prolific source, and, being situated so near to the rice tracts of the Cauvery delta, should naturally form the most important supply of phosphatic manure to the Tanjore District.

In what form or forms the nodules are to be applied is the question. Dr. Voelcker reported 32 years ago that it would not be remunerative to export them to foreign market for superphosphate manufacture, and my experiments go to confirm that it will not pay to manufacture superphosphate even in this country.

Analysis of phosphatic nodules.

These nodules contain 25 to 27 per cent. of phosphoric acid, equal to 56 to 59 per cent. of tricalcic phosphate, and are comparable to the fossiliferous South Carolina phosphates which contain 22 to 28 per cent. of phosphoric acid and to the coprolites of Cambridge which contain about 26 per cent. Closely associated as these nodules are with calcareous nodules and

* Since this memoir was sent to the press, Mr. V. S. Sambasiva Ayyar of Bangalore has reported the existence of some apatite beds in the Vizianagram Zemindary.

[1] Seaborne Trade of British India for the year 1920, p. 79.

[2] Soil Survey of the Tanjore District. *Madras Agri. Bull.*, No. 68, 1914.

gypsum, the flour phosphate sold by manure firms contains from 19 to 24 per cent. of phosphoric acid, several samples dealt with in this Laboratory containing 22 per cent.

These phosphatic nodules have been analysed, at the instance of Government, or for manure firms or private parties, in earlier years, and some of these analyses are found tabulated in Table I, along with the analysis made by the author.

TABLE I.

Analysis of phosphatic nodules of Trichinopoly.

Constituents	Voelcker, London 1892	King, Edinburgh 1892	Hooper, Madras 1893	Ramaswami Sivan, Coimbatore 1916
Moisture	1·53	0·94	1·53	1·22
Organic matter and water of combination	2·85	4·86	2·66	3·20
Insoluble matter	5·28	2·80	8·50	8·52
Phosphoric acid	26·05	27·04	24·58	25·63
Lime	45·51	46·88	43·51	42·06
Ferric oxide	3·14	3·96	8·62	2·58
Alumina	2·63			4·52
Magnesia	0·34	3·22	0·29	1·16
Alkalies and Fluorine	4·84		2·47	1·72
Sulphuric acid	0·60		0·50	0·45
Carbonic acid	7·23	10·30	7·38	8·94
TOTAL ..	100	100	100	100
EQUAL TO CALCIUM PHOSPHATE ..	56·87	59·00	53·65	55·95
EQUAL TO CALCIUM CARBONATE ..	16·43	23·41	16·77	17·41

The actual forms of combination in which the bases and acids are present in the complex nodule may be different in the different samples; and, for purposes of calculation, these are assumed to contain, in the sample analyzed by the author, 55·95 parts of tricalcium phosphate, 17·41 parts of calcium carbonate, 2·44 parts of magnesium carbonate, and iron oxide and alumina, either free or as silicate. Every 100 parts by weight of nodules would then

require 35·44 parts of sulphuric acid to convert the tricalcic phosphate into mono-calcic phosphate and 37·68 parts to dissolve the carbonates, iron oxide and alumina. In other words, as much sulphuric acid is wasted in reacting with the impurities as in the conversion of the insoluble phosphate into a soluble form. At the price at which sulphuric acid can be procured in Madras—there is only one sulphuric acid factory in the Presidency at Ranipet—superphosphate cannot be manufactured out of the nodules at a cost less than Rs. 16 per unit of phosphoric acid, while the manure firms in Madras are now selling bonechar superphosphate at Rs. 7 to Rs. 8 per unit. The high percentage of calcium carbonate (17 per cent.) and of iron oxide and alumina (7 per cent.) detract from the value of the nodules for superphosphate manufacture, and the stuff cannot be converted into super until chamber acid sells at 9 pies per pound. This explains why so many firms who have been interesting themselves in these deposits from 1892 up to date had to give up the idea of working them.

The manufacture of super from the nodules being out of the question, in what other ways can they be utilized ? Within the last few years, an electrolytic method,[1] worked on a small factory scale in Sweden, has been suggested whereby even low grade phosphates can be converted into dicalcic phosphate easily soluble in soil water. The question of developing a hydro-electric scheme, from the water power available in the adjacent Pachamalai and Kollemalai Hills, was once and, it is believed, is even now seriously under consideration; but, until cheap electric supply is available, the manufacture of the nodules into dicalcic phosphate at the locality need not be considered.

There is then only one alternative left, and that is to utilize the nodules in a powdered form as flour phosphate directly as a manure—a recommendation made by Dr. Voelcker 25 years ago.

2. Review of literature on the use of raw phosphate.

Earlier trials[2] with the powdered phosphate in some of the coffee plantations in Southern India and Ceylon seem to have been disappointing, while Hooper states that it gave increased crops with a few plants in the Nilgiris.[3] Flour phosphate has given as good a yield as bonemeal with paddy in

[1] *Chemical News*, 29th August, 1913, paper by Palmaer at the Eighth International Congress of Applied Chemistry.

[2] *Rec. Geo. Sur. India*, Vol. XXXIX, 1910, p. 51;

[3] *Agricultural Ledger*, 1898, No. 20, pp. 16–19.

Manganallur Farm.[1] Burlison of Illinois State[2] recommends the direct application of powdered rock phosphate along with organic matter in the form of green manure or cattle manure. Cameron and Bell,[3] speaking of South Carolina phosphates—which are similar in composition to the nodules—show that the solubility and decomposition of rock phosphates are much increased and hastened by the presence of carbonic acid in water and that, when organic matter, especially green manure, is thoroughly incorporated in the soil with the phosphates, the carbonic acid and possibly other acids resulting from the decomposition of the organic matter so increase their solubility as to make them compare favourably with the rapidly soluble phosphates.

Since the publication of the preliminary account of the subject of this memoir in the Madras *Year Book*, there has been a vast addition to the literature on the use of raw phosphate ; and mention might advantageously be made here to a paper[4] which almost summarizes the present position with regard to the efficacy of mineral phosphate as a phosphatic manure.

Albert F. Ellis, New Zealand Phosphate Commissioner, who has used finely ground mineral phosphate in the place of basic slag and superphosphate with great success, elicited the opinions of several scientists in Great Britain and the United States of America regarding the manurial action of mineral phosphate, some of which are as follows :—

New Zealand experience. On soils with a sour tendency or fairly rich in humus and in districts where the rainfall is satisfactory, finely ground raw phosphate gives good results, particularly when used for top-dressing.

British experience. Dr. E. J. Russell, Director, Rothamsted Station, endorses the New Zealand experience. In his "Manuring for Crop Production, 1917," he states that mineral phosphates give good results when ground sufficiently fine ; that, when immediate action is necessary, they may not help the young plant, being in this respect inferior to superphosphate and basic slag, but that the difficulty may be overcome by mixing with a certain proportion of superphosphate, whereby the seedling stage is assisted and the older plant can then make use of the phosphate : and that, in the warmer and moister region of North Wales, the superphosphate is unnecessary and the phosphate by itself gives satisfactory increases.

[1] *Madras Agri. Dept. Bull.* No. 85.

[2] Burlison, W. L. (Illinois). Availability of mineral phosphates for plant nutrition. *Jour. Agri. Res.*, Vol. VI, No. 13, 1916, pp. 485–514.

[3] *United States, Bureau of Soils, Bulletin* No. 41, 1907.

[4] *New Zealand Jour. Agri.*, Vol. XXII, No. 6, 1921.

Professor James Herdrick of the College of Agriculture, Aberdeen, states that his results agree with those obtained in New Zealand, the soils referred to by him being deficient in lime, rich in humus and tending to be sour. Commenting on the results of some field experiments,[1] he arrives at the conclusions (i) that the general effect of superphosphate is only slightly greater than that of insoluble phosphate, when equal quantities of phosphoric acid are applied, (ii) that, if one-third of the phosphoric acid is given as soluble phosphoric acid, and the remainder as insoluble phosphate, the average result is as good as when the whole of the phosphoric acid was given as a soluble phosphate, (iii) that the soluble phosphate was hitherto overvalued and insoluble phosphate undervalued, and (iv) that, in future, more of the rock phosphate in finely ground condition should be used in farming, thereby saving acid and expense.

Professor Douglas A. Girlchirst of the Armstrong College, Newcastle-on-Tyne, writes that mineral phosphates, when ground as finely as basic slag, are equally effective, that they deserve more attention from the experimenter and the farmer, that the cost per unit is considerably less than in basic slag, and that Tunisian and Belgian phosphates have given quite satisfactory results.

Dr. S. G. Robertson of the East Anglian Institute of Agriculture, Chelmsford, writes that his experience agrees with the New Zealand results, in that, on sour soils, well supplied with organic matter and where the rainfall is high—about thirty inches—there is little to choose between the different rock phosphates and the best types of basic slag, that, when the soil is sweet and the rainfall is as low as twenty or twenty-two inches, they are not as effective as superphosphate or the best grades of basic slag, and that, where the soil is deficient in lime, the rock phosphates give more satisfactory results than superphosphates.

United States experience. In the State of Illinois, the Agricultural Experiment Station has been carrying on vigorous propaganda in favour of finely ground rock phosphate, not on account of its immediate results, but as being conducive to permanent soil fertility. The farmers of Illinois corn-belt find that rock phosphate gives good stands of clover and other legumes, and that improvement in crops following the legumes is evident everywhere.

Apart from Illinois, several other States seem to have had similar experience with rock phosphate, in districts where the soils are sour and rich in organic matter.

[1] *Jour. Soc. Chem. Indus.*, 1919, Vol. XXXVIII, No. 9, p. 157 R

Waggman and Wagner of Wisconsin recently compiled a publication[1] dealing with the use of raw phosphate in all the States of America and, while the mass of evidence is in its favour, the summary of results is rather inconclusive. With a view to determine the sentiment towards ground raw phosphate as a fertilizer, a letter and a set of questions were sent to 1,000 progressive farmers who had used the material. Out of 315 replies received, 219 farmers or 69·6 per cent. were favourable to its use, 55 or 17·5 per cent. were doubtful about its action and 41 or 13 per cent. regarded the material unfavourably.

Reviewing the experimental work with raw phosphate in all the States of the United States of America, they arrive, among others, at the following conclusions :—

(1) The conventional laboratory methods so far proposed for determining the availability of phosphoric acid in various phosphates, while of some use, do not necessarily serve as an index to its availability under soil conditions.

(2) The application of liberal and even medium quantities of raw phosphate to most soils produces an increase in the yields of many crops in the first year.

(3) The effectiveness of raw phosphate depends largely on its thorough distribution in the soil, the distribution being brought about by liberal applications of very finely divided material and thorough cultivation.

(4) The presence of decaying organic matter in the soil increases the effectiveness of ground raw phosphate, owing probably both to greater bacterial activity and the higher content of carbonic acid in such soils.

(5) As a corollary of 3 and 4, the effectiveness of phosphate is usually increased after remaining in the soil for a year or more.

(6) Most crops respond more quickly to acid phosphate than to bone, basic slag or raw phosphate. Therefore, when early stimulation and quick maturity of crop are the main considerations, acid phosphate is the best form of phosphoric acid to apply.

(7) The question which will be more economical to use to produce an increase in yield must be considered separately in each case, the factors affecting being the nature of the soil, the crop system

[1] *United States, Bureau of Soils, Bulletin* No. 699, 1918, p. 113.

employed, the price of the different phosphates and the length of the growing season.

3. Lines of investigation.

A phosphatic manure is required for the paddy soils of Madras, which receive, however, a different cultural treatment from the soils of Europe and America. It was considered desirable, therefore, to determine, by actual experiments, how far flour phosphate, by which name the ground phosphatic nodules are known in the Presidency, is a suitable manure for paddy *under the swampy conditions of cultivation* prevailing in this country. Experiments were carried out for some years *to determine the availability of flour phosphate* for such purposes in the following directions :—

(*a*) By measuring its solubility in carbonic acid.
(*b*) By measuring its solubility in citric acid and ammonium citrate.
(*c*) By measuring its solubility in composts made with green manure or cattle manure.
(*d*) By the growth of paddy in pots, in conjunction with green manure or cattle manure.
(*e*) By the growth of paddy in pots, with and without green manure, but with increasing quantities of phosphate.
(*f*) By the growth of paddy in pots, with and without green manure, and with increasing quantities of phosphate, nitrogen being also supplied.
(*g*) By the growth of paddy on field scale in co-operative experiments on ryots' lands and in Government Agricultural Stations, with green manure and phosphate as against green manure only.

These experiments will be discussed in detail in sections (*a*) to (*g*).

(*a*) Solubility of flour phosphate in carbonic acid.

Material used. Selected phosphatic nodules were crushed and pounded in an agate mortar until the whole passed through the hundred-mesh sieve.

First method. Four grm. of the phosphate were suspended in 250 c.c. of water in a measuring cylinder and carbonic acid was passed through from a Kipp's apparatus for 15 minutes, the gas being purified by bubbling it previously through water. The solid matter was allowed to settle, and the supernatant liquid decanted over a dry filter. Phosphoric acid was estimated, in duplicate, in aliquot parts of the filtrate, the filter paper was thoroughly

washed into the same cylinder, water was added to the 250 c.c. mark, carbonic acid was passed as before and the filtrate examined for phosphoric acid. The above processes were repeated ten times, and the final residue, together with the ash of all the filter papers, was examined for insoluble phosphoric acid. Simultaneously 2 grm. of pure tricalcic phosphate were similarly treated. The results are shown in Table II.

TABLE II.

Showing the action of carbonic acid in dissolving flour phosphate and phosphate of lime.

Extractions	Weight of phosphoric acid in 250 c.c. of extract	
	Flour phosphate	Pure phosphate of lime
	grm.	grm.
1st extraction	0·0415	0·0510
2nd ,, ...	0·0351	0·0415
3rd ,, ...	0·0319	0·0475
4th ,, ...	0·0383	0·0606
5th ,, ...	0·0255	0·0383
6th ,, ...	0·0287	0·0415
7th ,, ...	0·0383	0·0351
8th ,, ...	0·0319	0·0510
9th ,, ...	0·0446	0·0415
10th ,, ...	0·0383	0·0351
TOTAL DISSOLVED IN 10 EXTRACTIONS	0·3541	0·4431
PHOSPHORIC ACID IN RESIDUE AFTER 10 EXTRACTIONS ..	0·6754	0·3955
TOTAL PHOSPHORIC ACID (ACTUAL)	1·0295	0·8386
,, ,, ,, (THEORETICAL)	1·0250	0·8400

The irregular variation in the quantity of phosphoric acid dissolved in successive extractions may be due to several causes, one of the most apparent being the different strength of carbonic acid generated each time, as the Kipp's apparatus did not work uniformly throughout the ten extractions nor throughout the 15 minutes' duration of each extraction. Taking the average, however, it is found that 0·3541 grm. of phosphoric acid equivalent to 0·773 grm. of tricalcic phosphate is dissolved out by 2,500 c.c. of water through which carbonic acid is repeatedly passed, so that one gram of tricalcic phosphate is soluble in 3,234 c.c. of carbonic acid water.

As regards pure tricalcic phosphate, 0·4431 grm. of phosphoric acid, equivalent to 0·9673 grm. of the phosphate, is dissolved in 10 extractions,

so that one gram of pure tricalcic phosphate is soluble in 2,584 c.c. of carbonic acid water.

2nd method. Two grm. of flour phosphate were shaken for 15 minutes with 500 c.c. of water saturated with carbonic acid at room temperature (27°C.) and the filtrate was examined as before. 0·0295 grm. of phosphoric acid equivalent to 0·0644 grm. of tricalcic phosphate is found soluble in 500 c.c. of carbonic acid water, so that one gram of tricalcic phosphate as present in flour phosphate is soluble in 7,764 c.c. of carbonic acid water.

The difference in the solubility of the phosphate in the two methods is due to the fact that, in the first method, repeated quantities of carbonic acid continued to act on the material, while in the second, there was only a limited quantity of carbonic acid in the solution. Moreover, as the material contains much calcium carbonate, a greater amount of soluble calcium bicarbonate will be produced in the first method, and the bicarbonate is well established as a carrier of carbonic acid.

Solubility as determined by different workers.

The solubility of tricalcic phosphate in various forms as determined by different chemists is given below[1] for comparison :—

TABLE III.

Nature of phosphate	Parts of carbonic acid water required to dissolve 1 part of phosphate	By whom determined	REMARKS
Precipitated tricalcic phosphate	1,789	Warrington	50°F & 750 mm.
„ „ „	1,102	Bischoff	
„ „ „	1,333	Lassaigne	
Dry „ „	12,591	Voelcker	
Dry, ignited „ „	31,818	„	
Dry phosphate in apatite	222,000	Williams	
Levigated „ „	140,000	„	
Phosphate in bone, finely ground ..	5,698	„	
„ „ burnt	8,029	„	
„ „ dust	4,122	„	
„ in South Carolina phosphate ..	6,983	„	
„ „ „ levigated ..	6,544	„	
„ in guano	8,009	„	
Phosphate in phosphatic nodules, in water saturated with carbonic acid ..	7,764	Ramaswami Sivan	27°C. & 725 mm.
„ in phosphatic nodules, acted upon by carbonic acid for 15 minutes at a time, repeatedly ..	3,234	„	
Pure tricalcic phosphate	2,584	„	

[1] Storer. *Agriculture in some of its relations with Chemistry*, 1906, Vol. 1, p. 444.

From the above table it is seen that tricalcic phosphate in the phosphatic nodules is very much more soluble in carbonic acid than that in apatite. Its solubility is about the same as Carolina phosphate, with which, as stated above, it is largely comparable.

Qualitatively the solvent action of carbonic acid upon tricalcic phosphate is demonstrated by shaking the phosphate with distilled water in a test tube and examining the filtrate for phosphoric acid, when little or no precipitate will be produced with ammonium molybdate, whereas, if carbonic acid be passed into the test tube for a few seconds, the filtrate will show an appreciable quantity of the yellow phospho-molybdate.

Schlœsing has shown *quantitatively* that tricalcic phosphate is increasingly soluble in water containing increased quantities of carbonic acid, while its solubility in pure water is very slight, as will be seen from Table IV.[1]

TABLE IV.

Showing solubility of phosphate of lime in increasing quantities of carbonic acid.

Solvent	Phosphoric acid dissolved per litre in mg.
Water	0·74
1,200 c.c. of water + 50 c.c. of water saturated with carbonic acid ..	6·90
1,000 c.c. of water + 250 c.c. of water saturated with carbonic acid ..	48·50
1,250 c.c. of water saturated with carbonic acid	91·90

It is evident, therefore, that, to make the tricalcic phosphate in the powdered nodules a quick acting manure, the production of large quantities of carbonic acid should be induced. The larger the quantity of carbonic acid reacting on the phosphate, the greater will be its solubility.

Quantities of carbonic acid produced in soils.

Hutchinson and Milligan,[2] in examining the gases formed in Pusa soil, state that, while only 91·5 milligrams of carbonic acid are produced in sixteen days in 16 litres of air aspirated from untreated soil, 939·4 milligrams are

[1] *U. S. A., Bureau of Soils, Bulletin* No. 41.

[2] Hutchinson, C. M., and Milligan, S. Green Manuring Experiment, 1912-13. *Agri Res. Inst., Pusa, Bull.* 40, p. 30.

produced in a soil containing 16 per cent. moisutre to which green manure was added. This works out to 430 lb. of carbonic acid produced in sixteen days per acre to six inches depth, assuming that 50 per cent. is the total pore-space in the soil.

The action of carbonic acid on tricalcic phosphate is generally accepted as taking place according to either of the following equations :—

$$Ca_3P_2O_8 + CO_2 + H_2O = Ca_2H_2P_2O_8 + CaCO_3 \text{ ; or}$$
$$310 \qquad 44$$

$$Ca_3P_2O_8 + 2CO_2 + 2H_2O = CaH_4P_2O_8 + 2CaCO_3.$$
$$310 \qquad 88$$

According to Hutchinson and Milligan, therefore, the amount of carbonic acid (430 lb.) produced in arable soil is sufficient to convert 3,030 lb. of tricalcic phosphate into dicalcic phosphate or 1,515 lb. of tricalcic into monocalcic phosphate.

But a phosphatic manure is wanted for paddy soils ; and the Memoirs on paddy soil gases published by Harrison and Subramania Ayyar[1] show that, in the initial stages of fermentation of green manure, 5 to 20 per cent. of the gases may consist of carbonic acid, that, under the anærobic conditions prevailing, marsh gas and hydrogen are prominent in later stages, that the marsh gas is oxidised by the methane-oxidising bacterium, and that the resulting carbonic acid is decomposed by the film algæ liberating oxygen. The following experiment was, however, devised to determine how far the carbonic acid produced in swampy soil would be sufficient to dissolve the phosphate.

To a large pot containing 8,000 grm. of paddy soil to a depth of six inches, 50 grm. of fresh *dhaincha* plants cut in small pieces were mixed and the soil was puddled with water, with a layer of half inch of water above the puddle. A twelve-inch diameter funnel was inverted on the soil, and the stem of the funnel was provided with a double-hole cork, through which passed two glass tubes, the ends of which did not quite touch the level of the water in the pot. The outer end of one of the glass tubes was attached to an absorption bottle containing caustic soda, while the end of the other glass tube was attached to another absorbing bottle containing 100 c.c. of N/10 barium hydroxide, which, in its turn, was attached to a large aspirating bottle (Fig. 4). Once in 24 hours, 13 litres of air were aspirated through the whole apparatus, the barium hydrate bottle being removed and replaced by another similar

[1] Harrison, W. H. and Subramania Ayyar, P. A. The Gases of Swamp Rice Soils. *Mem. Dept. Agri. India, Chem. Ser.*, Vol. III, No. 3. Vol. IV, Nos. 1 and 4; Vol. V, Nos. 1, 7 and 8.

bottle every day. The quantity of carbonic acid produced daily is shown in Table V.

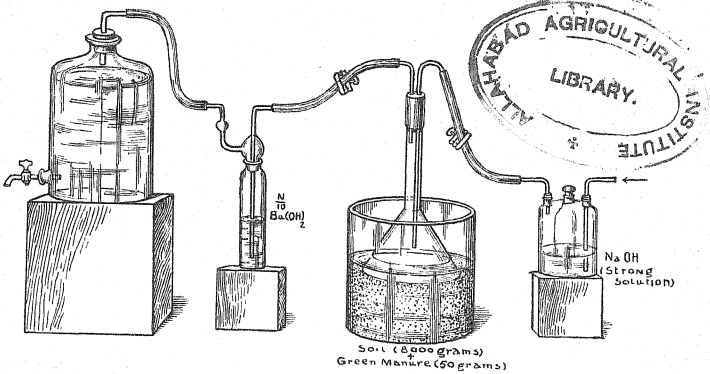


FIG. 4. Apparatus to determine the quantity of CO_2 generated in soils puddled with green manure.

TABLE V.

Showing quantity of carbonic acid aspirated in 16 days from soil in which green manure had been puddled.

Day	Weight of carbonic acid aspirated in 13 litres in grm.
1st	0·150
2nd	0·049
3rd	0·028
4th	0·050
5th	0·072
6th	0·057
7th	0·105
8th	0·116
9th	0·172
10th	0·210
11th	0·251
12th	0·221
13th	0·220
14th	0·167
15th	0·107
16th	0·082
TOTAL FOR 16 DAYS	2·057 grm.

It has to be mentioned that, with a view not to aerate the soil and in order to preserve anærobic conditions within the puddle, the glass tubes inside the stem of the funnel through which air was aspirated did not reach the level of the water in the pot. Moreover, no account is taken of the carbonic acid dissolved in the soil water or entangled in the soil mass, but only that portion of the gas evolved into the atmosphere above the water level. The quantity not taken into consideration must be considerable, for, on the first day after the experiment was started, the amount of carbonic acid aspirated was 0·150 grm. while the amounts on subsequent five days were much smaller, from 0·049 grm. to 0·057 grm. From the seventh day, the quantities measured were greater. The fermentation of green manure is inferred to be fairly rapid in the beginning, and the initial productions of carbonic acid must have been dissolved in the soil water or occluded within the soil mass. For purposes of calculation, the quantities of carbonic acid produced during the first sixteen days have alone been taken into consideration. Now 208 litres aspirated during these sixteen days contained 2·057 grm. of carbonic acid. An acre of paddy soil, six inches deep, with an apparent specific gravity of 1·20 weighs about 1,633,500 lb. As 8,000 grm. of soil, to which green manure was added, gave 2·057 grm. of carbonic acid, an acre of land to which similar proportion of green leaf is applied would give 420 lb. of carbonic acid, which would be sufficient to convert 2,959 lb. of tricalcic into dicalcic phosphate or half the quantity into monocalcic phosphate.

The decomposition of green leaves in puddled soil will produce not only carbonic acid, but various organic acids which go to make up sourness in soils. Even when these acids are not taken into consideration, the carbonic acid produced in swampy soils is sufficient to account for the solubility of phosphates required for plant nutrition.

(*b*) Solubility of flour phosphate in citric acid and other reagents.

Various strengths of different reagents have been recommended by chemists from time to time—some of them being termed official methods—for the estimation of available phosphoric acid in *soils* and *manures*. One per cent. citric acid is the one usually adopted for the former, while neutral ammonium citrate, two per cent. citric acid, one per cent. citric acid and N/10 citric acid have been recommended for the latter. In soils and in manures containing calcium carbonate, some of the citric acid, if not the whole, will be neutralized by the lime, and the actual reagent taking part in the reaction is chiefly calcium citrate. Table VI shows the relative solubility

of flour phosphate in different reagents, two grammes of the substance being heated in each case for half an hour at 65°C. with 100 c.c. of the reagent.

TABLE VI.

Showing solubility of flour phosphate in citric acid and ammonium citrate.

Nature and strength of reagent	Percentage of phosphoric acid rendered soluble	Ratio of soluble phosphoric acid to total phosphoric acid
2 per cent. citric acid ..	1·575	6·2
1 „ „	0·950	3·7
0·5 „ „	0·525	2·1
0·25 „ „	0·313	1·2
0·1 „ „	0·153	0·6
N/10 „ „	0·740	2·9
Neutral ammonium citrate ..	0·760	3·0

The amount of phosphoric acid rendered soluble by ammonium citrate is found to vary with the temperature, being greater in the cold than when the reagent is heated, for which information the author is indebted to Dr. Harrison.[1] 3·85 per cent. of the total phosphoric acid is rendered soluble when shaken for half an hour in the cold, as against 3·0 per cent. when heated at 65°C.; and this is apparently due to some of the calcium citrate being precipitated during the heating.

(*c*) SOLUBILITY OF FLOUR PHOSPHATE IN COMPOSTS.

A number of experiments were designed from time to time, from 1915 onwards, to determine the amount of phosphoric acid rendered soluble, as a result of composting the phosphate with green manure and cattle manure, with and without paddy soil. Reagents used to dissolve the phosphate and the temperature of the reaction were also changed, as suggested by experience. The materials used, the methods of composting and the methods of estimation will be described in detail.

Materials used.

Green manure. Whole full grown *dhaincha* (*Sesbania aculeata*) plants just in bloom were cut up into small bits, dried in air, and ground in a mortar until the whole passed through 1 mm. sieve.

Cattle manure. Rotten farmyard manure from one of the loose boxes of the Central Farm was dried in air and ground until the whole passed through 1 mm. sieve.

Soil. Paddy soil from M Block of the Central Farm was dried in the air and powdered until the whole passed through 1 mm. sieve.

[1] Harrison, W. H., and Das, S. L. The retention of soluble phosphates in calcareous and non-calcareous soils. *Mem. Dept. Agri. India, Chem. Ser.*, Vol. V, No. 9, p. 203.

Explanatory notes for Tables VII and VIII.

Column No. 1. Serial number of glazed earthenware pots containing the composts.

Column No. 2. P=Flour phosphate; G=Green manure; C=Cattle manure; the figures in brackets represent weights in grm.

Column Nos. 3, 4 and 5. From the moisture determination of each sample, the total dry weight is calculaetd (column 5), the dry weight of phosphate is taken to be constant (column 3), and the difference is shown in column 4 as the dry weight of green manure or cattle manure.

Column Nos. 6, 7 and 8. Figures in column 8 represent the weight in grm. of phosphoric acid actually determined in each compost and these were found to agree with theoretical values generally, while those in column 6 show the phosphoric acid present in the flour phosphate pots in each compost. The difference between these two figures is shown as total phosphoric acid derived from the decomposing organic matter.

Column Nos. 9, 10 and 11. Figures in column 11 represent the weight in grm of phosphoric acid soluble in 1 per cent. citric acid in each compost, while those in column 10 show the amount of phosphoric acid of the green manure or cattle manure rendered soluble, the figures for each compost being calculated from the compost of green manure only in each series. The difference between columns 11 and 10 is taken to be the amount of phosphoric acid of the flour phosphate rendered soluble (column 9).

Column No. 12. The ratio of soluble phosphoric acid of the flour phosphate shown in column 9 to the total phosphoric acid in it shown in column 6 is expressed as a percentage in column 12.

TABLE VII.

Showing solubility of flour phosphate (P), as a result of composting with green manure (G), measured by shaking with 1 per cent. citric acid for half an hour.

No.	Nature of compost	Calculated dry weight of compost			Total phosphoric acid in compost			Soluble phosphoric acid in compost			Percentage of soluble phosphoric acid in flour phosphate to total phosphoric acid
		P	G	Total	P	G	Total	P	G	Total	
1	2	3	4	5	6	7	8	9	10	11	12
	Original mixture	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	Percentage
1	P (100) ..	98·98	..	98·98	22·20	..	22·20	0·850	..	0·850	3·8
3	G (100) ..	..	91·97	91·97	..	0·71	0·71	..	0·57	0·57	....
7	P (50) & G (50)	49·49	46·86	96·35	11·10	0·36	11·46	1·02	0·29	1·31	9·0
8	P (20) & G (80)	19·80	73·77	93·57	4·44	0·57	5·01	0·89	0·46	1·35	20·1
9	P (10) & G (90)	9·90	83·42	93·32	2·22	0·64	2·86	0·67	0·51	1·18	30·2
	Fifteen days' compost										
12	G (100) ..	..	74·14	74·14	..	0·69	0·69	..	0·55	0·55	....
16	P (50) & G (50)	49·49	34·18	83·67	11·10	0·35	11·45	0·49	0·28	0·77	4·1
17	P (20) & G (80)	19·80	57·17	76·97	4·44	0·55	4·99	0·51	0·44	0·95	11·6
18	P (10) & G (90)	9·90	63·17	73·07	2·22	0·62	2·84	0·43	0·49	0·92	19·4
	One month's compost										
21	G (100) ..	..	55·71	55·71	..	0·72	0·72	..	0·57	0·57	....
25	P (50) & G (50)	49·49	30·27	79·76	11·10	0·36	11·46	0·47	0·29	0·76	4·2
26	P (20) & G (80)	19·80	48·08	67·88	4·44	0·58	5·02	0·24	0·45	0·69	5·4
27	P (10) & G (90)	9·90	52·52	62·42	2·22	0·65	2·87	0·28	0·51	0·79	12·6
	Three months' compost										
30	G (100) ..	..	49·35	49·35	..	0·70	0·70	..	0·54	0·54	....
34	P (50) & G (50)	49·49	27·94	77·43	11·10	0·35	11·45	0·44	0·27	0·71	4·0
35	P (20) & G (80)	19·80	36·65	56·45	4·44	0·56	5·00	0·20	0·43	0·63	4·5
36	P (10) & G (90)	9·90	40·72	50·62	2·22	0·63	2·85	0·27	0·48	0·75	12·1
	Six months' compost										
39	G (100) ..	..	41·76	41·76	..	0·66	0·66	..	0·56	0·56	....
43	P (50) & G (50)	49·49	25·87	75·36	11·10	0·33	11·43	0·50	0·28	0·78	4·5
44	P (20) & G (80)	19·80	25·59	45·39	4·44	0·53	4·97	0·31	0·45	0·76	7·2
45	P (10) & G (90)	9·90	34·94	44·84	2·22	0·59	2·81	0·19	0·50	0·69	8·5

Method of making composts and general remarks about the composts.

Table VII. Flour phosphate and green manure in the proportions shown in the Table were mixed and placed in small glazed earthenware pots, water was added enough to soak the mixture which was then covered with glass plates. Whenever the surface of the compost became dry, which was the case chiefly in long-kept composts, water was added from time to time to keep the surface moist. The decomposition of organic matter was rapid and great, as is seen from the diminution in total dry weight of green manure in each series (column 4). In some of the long-kept composts, worms developed. The composts were not stirred.

TABLE VIII.

Showing solubility of flour phosphate (P), as a result of composting with cattle manure (C), measured by shaking at 65°C. with 1 per cent. citric acid for half an hour.

No.	Nature of compost	Calculated dry weight of compost			Total phosphoric acid in compost			Soluble phosphoric acid in compost			Percentage of soluble phosphoric acid in flour phosphate to total phosphoric acid
		P	C	Total	P	C	Total	P	C	Total	
1	2	3	4	5	6	7	8	9	10	11	12
	Original mixture	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	Percentage
1	P (100) ..	98·98	..	98·98	22·20	..	22·20	0·85	..	0·85	3·8
2	C (100) ..	..	95·07	95·07	..	0·86	0·86	..	0·49	0·49	....
4	P (50) & C (50)	49·49	47·60	97·09	11·10	0·43	11·53	1·00	0·25	1·25	9·0
5	P (20) & C (80)	19·80	76·40	96·20	4·44	0·69	5·13	0·93	0·39	1·32	20·9
6	P (10) & C (90)	9·90	85·89	95·79	2·22	0·77	2·99	0·76	0·44	1·20	34·2
	Fifteen days' compost										
11	C (100) ..	..	94·38	94·38	..	0·86	0·86	..	0·56	0·56	....
13	P (50) & C (50)	49·49	44·35	93·84	11·10	0·43	11·53	0·88	0·28	1·16	7·9
14	P (20) & C (80)	19·80	72·88	92·68	4·44	0·69	5·13	0·68	0·45	1·13	15·4
15	P (10) & C (90)	9·90	82·17	92·07	2·22	0·77	2·99	0·55	0·50	1·05	24·8
	One month's compost										
20	C (100) ..	..	93·05	93·05	..	0·83	0·83	..	0·62	0·62	....
22	P (50) & C (50)	49·49	45·22	94·71	11·10	0·42	11·52	0·84	0·31	1·15	7·6
23	P (20) & C (80)	19·80	72·74	92·54	4·44	0·66	5·10	0·70	0·49	1·19	15·7
24	P (10) & C (90)	9·90	82·41	92·31	2·22	0·75	2·97	0·48	0·56	1·04	21·4
	Three months' compost										
29	C (100) ..	..	93·50	93·50	..	0·84	0·84	..	0·58	0·58	....
31	P (50) & C (50)	49·49	44·11	93·60	11·10	0·42	11·52	1·03	0·29	1·32	9·3
32	P (20) & C (80)	19·80	72·36	92·16	4·44	0·67	5·11	0·80	0·46	1·26	18·0
33	P (10) & C (90)	9·90	82·65	92·55	2·22	0·75	2·97	0·44	0·52	0·96	19·9
	Six months' compost										
38	C (100) ..	..	92·63	92·63	..	0·83	0·83	..	0·65	0·65	....
40	P (50) & C (50)	49·49	44·59	94·08	11·10	0·42	11·52	1·03	0·33	1·36	9·3
41	P (20) & C (80)	19·80	72·74	92·54	4·44	0·66	5·10	0·64	0·52	1·16	14·4
42	P (10) & C (90)	9·90	81·49	91·39	2·22	0·75	2·97	0·50	0·58	1·08	22·5

Table VIII. Flour phosphate and cattle manure in the proportions shown in the Table were mixed and placed in small glazed earthenware pots, water was added enough to soak the mixture which was then covered with glass plates. The surface of the composts did not get dry, to all appearance, as in Table VII, and similar quantities of water were not, therefore, added from time to time. The decomposition of organic matter was slow and the diminution in total dry weight of cattle manure was small in each series (column 4). In some of the long-kept composts, worms developed. The composts were not stirred.

Explanatory notes for Tables IX and X.

Column No. 1. Serial number of beakers containing the composts.

Column No. 2. S=Paddy soil which contained 0·0384 per cent. of total phosphoric acid and 0·0066 per cent. of available phosphoric acid. P=Flour phosphate—from a bulk sample from a manure firm. G=Green manure. The figures in brackets represent weights in grm.

Column Nos. 3, 4, 5 and 6. From moisture determinations of each sample, the total dry weight was calculated (column 6). The dry weights of soil and flour phosphate are taken to be constant in all the composts (columns 3 and 5). The dry weight of green manure shown in column 4 is obtained by difference.

Column Nos. 7, 8, 9 and 10. The figures in all these columns represent the theoretical values of phosphoric acid calculated from the quantities of each material put in the compost. The total weight of phosphoric acid shown in column 10 was also checked by actual determination.

Column Nos. 11, 12, 13 and 14. For purposes of calculation, the available phosphoric acid in the soil has been assumed to be the same in the composts as in the original soil (column 11), an assumption which is not quite correct and which should not have been made, were it not for the fact that the quantity of phosphoric acid in the soil used in the composts is very small. The available phosphoric acid due to the green manure in each compost shown in column 12 has been calculated from that of the green manure only in each series. The figures in column 14 show actual determinations, and those in column 13 are obtained by difference and represent the amount of phosphoric acid of the flour phosphate which has been rendered soluble in ammonium citrate at 65°C. or at room temperature as the case may be.

Column 15. The ratio of citrate soluble phosphoric acid of the flour phosphate shown in column 13 to the total phosphoric acid in it shown in column 9 is expressed as a percentage in column 15.

TABLE IX. *Showing solubility of flour phosphate (P), as a result of composting with soil (S) and green manure (G), measured by shaking with neutral ammonium citrate at 65°C. for half an hour.*

No.	Nature of compost	Calculated dry weight of compost				Total P_2O_5 in compost				Soluble P_2O_5 in compost				Percentage of citrate soluble phosphoric acid in flour phosphate to total P_2O_5
		S	G	P	Total	S	G	P	Total	S	G	P	Total	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
	Original mixture	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	Percentage
1	S (150) & P (100) ..	142·5	..	99·0	241·5	0·06	..	24·95	25·01	0·01	..	3·79	3·80	15·21
6	S (150) & G (100) ..	142·5	92·0	..	234·5	0·06	0·70	..	0·76	0·01	0·50	..	0·51	....
11	S (150) & G (50) & P (50) ..	142·5	46·0	49·5	238·0	0·06	0·35	12·48	12·89	0·01	0·25	3·16	3·42	25·28
16	S (150) & G (80) & P (20) ..	142·5	73·6	19·8	235·9	0·06	0·56	4·99	5·61	0·01	0·40	1·55	1·96	31·01
21	S (150) & G (90) & P (10) ..	142·5	81·8	9·9	235·2	0·06	0·63	2·50	3·19	0·01	0·45	0·86	1·32	34·52
	Seven days' compost													
2	S (150) & P (100) ..	142·5	..	99·0	241·5	0·06	..	24·95	25·01	0·01	..	4·29	4·30	17·18
7	S (150) & G (100) ..	142·5	86·7	..	229·2	0·06	0·70	..	0·76	0·01	0·48	..	0·49	....
12	S (150) & G (50) & P (50) ..	142·5	34·2	49·5	226·2	0·06	0·35	12·48	12·89	0·01	0·24	3·68	3·93	28·80
17	S (150) & G (80) & P (20) ..	142·5	76·6	19·8	238·9	0·06	0·56	4·99	5·61	0·01	0·38	2·46	2·85	49·35
22	S (150) & G (90) & P (10) ..	142·5	78·0	9·9	230·4	0·06	0·63	2·50	3·19	0·01	0·43	0·96	1·40	38·57
	Fifteen days' compost													
3	S (150) & P (100) ..	142·5	..	99·0	241·5	0·06	..	24·95	25·01	0·01	..	4·33	4·34	17·36
8	S (150) & G (100) ..	142·5	74·9	..	217·4	0·06	0·70	..	0·76	0·01	0·24	..	0·49	....
13	S (150) & G (50) & P (50) ..	142·5	32·2	49·5	224·2	0·06	0·35	12·48	12·89	0·01	0·48	2·66	2·91	21·29
18	S (150) & G (80) & P (20) ..	142·5	68·0	19·8	230·3	0·06	0·56	4·99	5·61	0·01	0·38	1·85	2·24	37·01
23	S (150) & G (90) & P (10) ..	142·5	76·3	9·9	228·7	0·06	0·63	2·50	3·01	0·01	0·43	1·10	1·54	43·89
	One month's compost													
4	S (150) & P (100) ..	142·5	..	99·0	241·5	0·06	..	24·95	25·01	0·01	..	4·31	4·32	17·26
9	S (150) & G (100) ..	142·5	81·7	..	224·2	0·06	0·70	..	0·76	0·01	0·50	..	0·51	....
14	S (150) & G (50) & P (50) ..	142·5	30·6	49·5	222·6	0·06	0·35	12·48	12·89	0·01	0·25	2·14	2·40	17·18
19	S (150) & G (80) & P (20) ..	142·5	56·8	19·8	219·1	0·06	0·56	4·99	5·61	0·01	0·40	1·45	1·86	29·14
24	S (150) & G (90) & P (10) ..	142·5	71·4	9·9	223·8	0·06	0·63	2·50	3·19	0·01	0·45	0·83	1·29	33·12
	Two months' compost													
5	S (150) & P (100) ..	142·5	..	99·0	241·5	0·06	..	24·95	25·01	0·01	..	4·29	4·30	17·21
10	S (150) & G (100) ..	142·5	66·1	..	208·6	0·06	0·70	..	0·76	0·01	0·51	..	0·52	....
15	S (150) & G (50) & P (50) ..	142·5	21·5	49·5	213·5	0·06	0·35	12·48	12·89	0·01	0·26	2·59	2·86	20·79
20	S (150) & G (80) & P (20) ..	142·5	46·7	19·8	208·0	0·06	0·56	4·99	5·61	0·01	0·41	1·38	1·80	27·74
25	S (150) & G (90) & P (10) ..	142·5	49·3	9·9	201·7	0·06	0·63	2·50	3·19	0·01	0·46	0·91	1·38	36·28

Table IX. Flour phosphate and green manure in the proportions shown in the Table, along with 75 grm. of soil, were mixed and placed in large beakers containing water enough to soak the compost, over which 75 grm. of soil were put in, and covered with glass plates. Water was added from time to time to keep the surface of the compost moist. The composts were not stirred and no worms developed in any of them. The decomposition of organic matter was fairly rapid (column 4).

TABLE X.

Showing solubility of flour phosphate (P), as a result of composting with soil (S) and green manure (G), measured by shaking with neutral ammonium citrate at room temperature, 27°C. to 28°C., for one hour.

No.	Nature of compost	Calculated dry weight of compost				Weight of total phosphoric acid				Weight of citrate soluble phosphoric acid in compost				Percentage of citrate soluble phosphoric acid in flour phosphate to total phosphoric acid
		S	G	P	Total	S	G	P	Total	S	G	P	Total	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
	Original mixture	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	grm.	Percentage
1	S (150) & P (50) ..	144·3	..	49·5	193·8	0·058	..	12·600	12·658	0·010	..	1·215	1·225	9·7
2	S (150) & G (50) ..	144·3	47·0	..	191·3	0·058	0·380	..	0·438	0·010	0·221	..	0·231	....
3	S (150) & P (25) & G (25) ..	144·3	23·5	24·75	192·55	0·058	0·190	6·300	6·548	0·010	0·1105	0·746[illegible]	0·867	11·9
4	S (150) & P (10) & G (40) ..	144·3	37·6	9·9	191·8	0·058	0·304	2·520	2·882	0·010	0·177	0·324	0·511	12·8
5	S (150) & P (5) & G (45) ..	144·3	42·3	4·95	191·55	0·058	0·342	1·260	1·660	0·010	0·199	0·210	0·419	16·7
	One week's compost													
6	S (150) & P (50) ..	144·3	..	49·5	193·8	0·058	..	12·600	12·658	0·010	..	1·474	0·484	11·7
7	S (150) & G (50) ..	144·3	35·8	..	180·1	0·058	0·380	..	0·438	0·010	0·359	..	0·369	....
8	S (150) & P (25) & G (25) ..	144·3	18·25	24·75	187·3	0·058	0·190	6·300	6·548	0·010	0·180	0·713	0·907	11·3
9	S (150) & P (10) & G (40) ..	144·3	29·3	9·9	183·5	0·058	0·304	2·520	2·882	0·010	0·287	0·416	0·713	16·5
10	S (150) & P (5) & G (45) ..	144·3	31·65	4·95	180·9	0·058	0·342	1·260	1·660	0·010	0·323	0·223	0·556	17·7
	Two weeks' compost													
11	S (150) & P (50) ..	144·3	..	49·5	193·8	0·058	..	12·600	12·658	0·010	..	1·513	1·523	12·0
12	S (150) & G (50) ..	144·3	32·7	..	177·0	0·058	0·380	..	0·438	0·010	0·351	..	0·361	....
13	S (150) & P (25) & G (25) ..	144·3	16·05	24·75	185·1	0·058	0·190	6·300	6·548	0·010	0·175	0·700	0·885	11·1
14	S (150) & P (10) & G (40) ..	144·3	26·8	9·9	181·0	0·058	0·304	2·520	2·882	0·010	0·281	0·392	0·683	15·5
15	S (150) & P (5) & G (45) ..	144·3	27·85	4·95	177·1	0·058	0·342	1·260	1·660	0.010	0·316	0·218	0·544	17·3
	Four weeks' compost													
16	S (150) & P (50) ..	144·3	..	49·5	193·8	0·058	..	12·600	12·658	0·010	..	1·468	1·478	11·7
17	S (150) & G (50) ..	144·3	29·8	..	174·1	0·058	0·380	..	0·438	0·010	0·319	..	0·329	....
18	S (150) & P (25) & G (25) ..	144·3	10·85	24·75	179·9	0·05	0·190	6·300	6·548	0·010	0·160	0·707	0·877	11·2
19	S (150) & P (10) & G (40) ..	144·3	21·4	9·9	175·6	0·058	0·304	2·520	2·882	0·010	0·255	0·364	0·629	14·4
20	S (150) & P (5) & G (45) ..	144·3	24·35	4·95	173·6	0·058	0·342	1·260	1·660	0·010	0·287	0·220	0·517	17·5

Table X. Flour phosphate and green manure in the proportions shown in the Table, along with 50 grm. of soil, were mixed and placed in large beakers containing water enough to soak the compost, over which 100 grm. of soil were put in, and covered with glass plates. Water was added from time to time to keep the surface of the compost moist. The composts were not stirred and no worms developed in any of them. The decomposition of organic matter was fairly rapid (column 4).

Examination of composts.

Sampling. At the end of the period of each compost, the whole contents were emptied into shallow dishes and quickly dried in the sun, and the drying was accelerated, when necessary, in the steam oven until the material was hard enough to be ground in a mortar when it was weighed and stored in bottles.

Estimation of moisture.

Five grm. of each stored sample were dried in the steam oven until weight was constant. From the moisture determination, the dry weight of each compost was calculated.

Estimation of total phosphoric acid. The phosphoric acid contained in each of the ingredients added to the compost having previously been ascertained, the theoretical quantity of phosphoric acid present in each compost is known. In addition, a direct estimation was made in each compost as follows :—

Two grm. were ignited, digested with hydrochloric acid, evaporated to dryness, ignited and digested with nitric acid, and the phosphoric acid was estimated in an aliquot of the nitric acid extract by precipitating with ammonium molybdate and titrating the washed phospho-molybdate with standard alkali, the work being done in duplicate right through. In the earlier stages of the investigation, owing to the author's inability to devote undivided attention to the work in progress, molybdic acid was apparently precipitated, and the actual determinations of phosphoric acid did not always tally with the theoretical values. Later on, however, the estimation was standardized by heating the solution to 65°C. in a water bath, adding freshly prepared mixture of nitric acid and ammonium molybdate and stirring continuously for 15 minutes at 65°C. over a water bath, whereby uniformly correct results were obtained in all cases.

Estimation of soluble phosphoric acid. Two grm. of the soil were digested with 100 c.c. of one per cent. citric acid at 65°C. for half an hour as regards composts shown in Tables VII and VIII, with 100 c.c. of neutral ammonium citrate (sp. gr. 1·09) at 65°C. for half an hour as regards composts shown in Table IX and with 100 c.c. of neutral ammonium citrate (sp. gr. 1·09) at room temperature (27° to 28°C.) for one hour with regard to composts shown in Table X. This was filtered and washed, and from the ignited filter the *citric acid-insoluble* or *citrate-insoluble* phosphoric acid, as the case may be, was estimated, the soluble phosphoric acid being found by difference, according to the usual method. In addition, the above filtrate was evaporated to

dryness, ignited and digested with nitric acid, and phosphoric acid was estimated in it. This determination formed a reliable check on the accuracy of the analysis.

The results of analysis are shown in detail in Tables VII, VIII, IX and X and represented in the charts in Figs. 5 and 6.

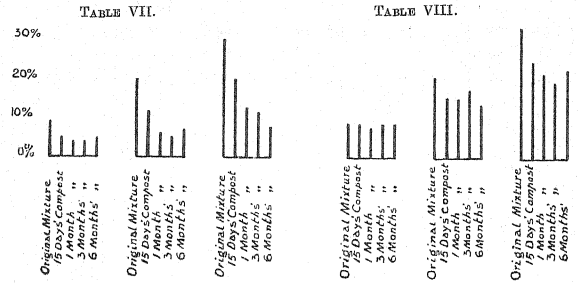


FIG. 5. Showing ratio of $\frac{\text{soluble } P_2O_5}{\text{total } P_2O_5}$ in composts with green manure and cattle manure.

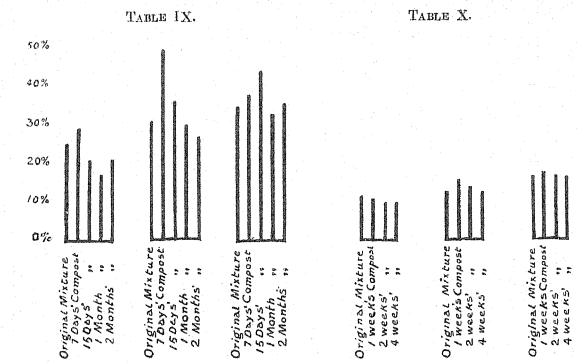


FIG. 6. Showing ratio of $\frac{\text{soluble } P_2O_5}{\text{total } P_2O_5}$ of Flour phosphate in composts made with green manure.

Discussion of results.

The original idea in starting these composting experiments was somewhat as follows :—

Organic matter in the form of green manure or cattle manure will, during its decomposition, evolve considerable quantities of carbonic acid and some organic acids as well. The greater the quantity of carbonic acid produced, the greater will the tricalcic phosphate contained in the phosphatic nodules be converted into dicalcic or monocalcic phosphate. Two methods can be suggested to effect this object, one to make the chemical reaction take place in the phosphatic manure previous to its application, and the other to induce the reaction to take place in the soil after application of the manure. The former can be carried out in practice in two ways, either by sprinkling daily a few lb. of flour phosphate on the cattle manure in the loose box or by piling up alternate layers of flour phosphate and green manure in compost pits : the latter can be adopted by applying the flour phosphate to the soil when green manure is puddled in.

Now let us consider the results obtained in the composting experiments shown in Tables VII to X.

Measured by treatment with 1 per cent. citric acid at 65°C., the percentage of soluble phosphoric acid in flour phosphate is greatest in the uncomposted original mixture and becomes less in each series of composts shown in Tables VII and VIII. As was stated above, decomposition of the organic matter was quicker and greater with green manure composts than with cattle manure composts. The non-addition of soil to these two classes of composts resulted in the development of worms whose life-growth might have interfered with normal chemical reactions in the compost heaps.

As regards composts shown in Tables IX and X, soil had been added, both to mix with the compost and to cover it, and no worms developed in any of them. Measured by treatment with neutral ammonium citrate at 65°C. or at room temperature, the percentage of soluble phosphoric acid in the flour phosphate was fairly large in the original uncomposted mixture and only slightly higher in one week's compost, remaining stationary or becoming slightly reduced again in longer kept composts, though these were not kept so long as the composts shown in Tables VII and VIII. In all the series of composts, however, it will be seen that the larger the proportion of organic matter with reference to the flour phosphate, the greater is the phosphate rendered soluble.

The comparatively smaller quantity of phosphate dissolved in Tables VII and VIII is apparently due to the fact that some, if not the whole, of the citric acid is used up in neutralizing the calcium carbonate which is present in the phosphatic nodules to the extent of 17 per cent., so that the reacting agent would be calcium citrate rather than citric acid. Heated for half an hour at 65°C., probably even some of this calcium citrate is precipitated and thrown out of the reaction.

From the large decrease in the dry weight of green manure in the different series of composts, it is inferred that considerable amount of decomposition has all along been going on in the composts and considerable quantities of carbonic acid and organic acids, which produce what is termed sourness or acidity in soils, must have been produced. One would naturally expect that the longer the compost was kept, the greater would be the amount of phosphate dissolved out of it. The results, however, are just the other way.

From the experiments detailed in Section 3(*a*) regarding the solvent action of carbonic acid on flour phosphate, it is presumed that, in longer kept composts, a greater amount of soluble phosphate is actually produced, but that this reacts with the lime present and gets reverted into insoluble phosphate. It has also to be remembered that the different laboratory methods of determining the availability of phosphates in soils and manures are far from satisfactory, but in the present case reversion of soluble phosphate is apparently the correct answer to the results obtained.

These results again indicate that there is something inherent in the green manure whereby the phosphate gives the maximum, or very nearly the maximum, amount of solubility in the uncomposted original mixture, and that possibly the extract obtained on digesting the green manure with different reagents would be sufficiently acid to dissolve the phosphate. To ascertain this point, the following experiment was done.

Experiment. Five grm. of green manure were shaken for half an hour with 100 c.c. of different reagents, some at room temperature and some at 65°C. as per Table XI. The extract was filtered over a dry filter and 25 c.c. of the filtrate were titrated with N/10 KOH, with phenolphthalein as indicator, with the following results :—

TABLE XI.

Showing the extent of acidity of green manure extracts with different reagents.

No. of experiment	Nature of experiment	Temperature	25 c.c. of extract required N/10 KOH	∴ 100 c.c. of extract = 5 grm. green manure N/10 KOH	REMARKS
			c.c.	c.c.	
1	Distilled water ..	28°C.	1·2	4·8	
2	,, ,, ..	65°C.	2·1	8·4	
3	Neutral ammonium citrate	28°C.	12·2	48·8	
4	,, ,, ..	65°C.	21·2	84·8	
5	Neutral ammonium citrate and $CaCO_3$	28°C.	1·2	4·8	$CaCO_3$ equivalent to 12·2 c.c. N/10 KOH (experiment 3).
6	,, ,, ..	65°C.	1·2	4·8	$CaCO_3$ equivalent to 21·2 c.c. N/10 KOH (experiment 4).
7	1 per cent. citric acid	28°C.	30·0	120·0	
8	,, ,, ..	65°C.	32·0	128·0	
9	1 per cent. citric acid and $CaCO_3$ ..	28°C.	6·5	26·0	$CaCO_3$ equivalent to 1 gramme of citric acid.
10	,, ,, ..	65°C.	5·3	21·2	

This simple experiment is interesting in its results. The solution obtained on digesting the green manure with distilled water is only slightly acid, while the acidity of the solution obtained on digesting with *neutral* ammonium citrate is great, especially at the temperature of 65°C. As the ammonium citrate reagent used in the experiment was distinctly neutral when titrated with N/10 KOH in a blank experiment, the acidity was developed in experiments 3 and 4, only after digestion with green manure, suggesting that the ammonia combined with the organic matter and liberated free citric acid. This supposition is confirmed in experiments 5 and 6, for, when equivalent quantities of calcium carbonate were added before digestion, the resulting solution behaved like the extract with distilled water in its power of neutralizing alkali. The reduced solvent action of 1 per cent. citric acid when equivalent quantities of $CaCO_3$ were added before digestion is again clearly shown in experiments 7 to 10.

Conclusion.

Once more the reactions which the phosphate undergoes in the soil may be expressed to occur according to the following four equations :—

$$Ca_3P_2O_8 + CO_2 + H_2O \longrightarrow Ca_2H_2P_2O_8 + CaCO_3 \quad \text{(Solution)} \quad \text{(i)}.$$

$$Ca_3P_2O_8 + 2CO_2 + 2H_2O \longrightarrow CaH_4P_2O_8 + 2CaCO_3 \quad \text{(Solution)} \quad \text{(ii)}.$$

$$Ca_2H_2P_2O_8 + CaCO_3 \longrightarrow Ca_3P_2O_8 + CO_2 + H_2O \quad \text{(Reversion)} \quad \text{(iii)}.$$

$$CaH_4P_2O_8 + 2CaCO_3 \longrightarrow Ca_3P_2O_8 + 2CO_2 + 2H_2O \quad \text{(Reversion)} \quad \text{(iv)}.$$

The reactions would be reversible and, in view of the comparatively large mass of lime present in the soil and in the flour phosphate, and comparatively small mass of carbonic acid generated under paddy soil conditions, the chemical changes would partake more of reversion of soluble into insoluble phosphate as shown in equations (iii) and (iv) than the other way.

Now, if the phosphate rendered soluble by decomposing organic matter gets reverted into insoluble phosphate, it is apparently better that the reversion should take place in the soil after application of the manure than that it should occur in the manure pit prior to application, for, in the former case, the reversion takes place round soil particles in a manner similar to the action of superphosphate, while, in the latter, the reverted phosphate of the manure has once again to be dissolved in the soil before plants can take it up. Conclusive results cannot, after all, be said to have been arrived at regarding the manurial efficiency of composts of flour phosphate and green manure, but it may be recommended that, with regard to paddy soils, flour phosphate is best applied along with green manure at the time of puddling.

(*d*) Availability of flour phosphate as measured by the growth of paddy plants in pots.

Experiments on the growth of the paddy plant in pots were carried on in the pot culture house for a number of years, and the results are given below :—

Pot Experiments—Series I (1916-17).

Soil used. Soil from the Manganallur Paddy Station was used for these experiments and had the following analysis :—

	Moisture	4·95
	Organic matter	5·83
	Insoluble silicates and sand	74·68
	Ferric oxide	5·07
	Alumina	6·70
	Lime	0·63
	Magnesia	0·51
	Potash	0·25
	Soda	0·29
	Carbonic acid	1·05
	Sulphuric acid	trace
	Phosphoric acid	0·04
	TOTAL ..	100·00
Containing	Nitrogen	0·063
„	Available potash	0·020
„	Available phosphoric acid	0·005

Method of experimenting.

8,000 grm. of dry soil, occupying six inches height in fourteen-inch pots, were poured in pots to which water had previously been added. Weighed quantities of flour phosphate, superphosphate, green manure and cattle manure were added to the puddle and thoroughly stirred, the pots being filled in duplicate as follows :—

No. of pot	Nature of experiment	
1 & 13	nil	No manure.
2 & 14	G	Green manure (*dhaincha*) at 5,000 lb. per acre
3 & 15	C	Cattle manure at 5,000 „ „ „
4 & 16	P	Flour phosphate at 250 lb. per acre (55·5 lb. P_2O_5)
5 & 17	P + G	P (250 lb.) & G (5,000 „) „ „
6 & 18	P + C	P (250 „) & C (5,000 „) „ „
7 & 19	S	Superphosphate at 340 „ „ „ (55·5 „ P_2O_5)
8 & 20	S + G	S (340 lb.) & G (5,000 „) „ „
9 & 21	S + C	S (340 „) & C (5,000 „) „ „
10 & 22	$\frac{P}{2}$ + $\frac{S}{2}$	P (125 „) & S (170 „) „ „
11 & 23	$\frac{P}{2}$ + $\frac{S}{2}$ + G	P (125 „) & S (170 „) & G (5,000 lb.) per acre
12 & 24	$\frac{P}{2}$ + $\frac{S}{2}$ + C	P (125 „) & S (170 „) & C (5,000 „) „ „

Robust paddy plants were planted single one week after the puddle was prepared, and gradually thinned until 5 plants were left in each pot. The pots were exposed to the sun during the day and moved into the shed at night and watered, from day to day, with rain water as required. The growth of the plants was watched and judged by the colour of leaves, the robustness of growth and the tillers produced. A few days before harvest, a number of photographs were taken. The dried plants were cut close to the soil, dried first in air and then in the steam oven and the dry weights of grain and straw noted (Table XII and Fig. 7).

TABLE XII.

Showing dry weight of paddy grain and straw in pot experiments.

G=5,000 lb. green manure; P=250 lb. flour phosphate=55·5 lb. phosphoric acid per acre;
C=5,000 lb. cattle manure; S=340 lb. superphosphate=55·5 lb. phosphoric acid per acre;
P/2=125 lb. flour phosphate per acre;
and S/2=170 lb. superphosphate per acre.

Nature of experiment	I				II				TOTAL OF I AND II			
	No. of pot	DRY WEIGHT OF			No. of pot	DRY WEIGHT OF			DRY WEIGHT OF			Relative yield of total dry produce (no manure as standard)
		Grain	Straw	Total		Grain	Straw	Total	Grain	Straw	Total	
		Grm.	Grm.	Grm.		Grm.	Grm.	Grm.	Grm.	Grm.	Grm.	
Nil ..	1	7·081	14·032	21·113	13	8·775	16·228	25·003	15·856	30·260	46·116	100
G ..	2	8·994	16·410	25·360	14	11·486	20·291	31·777	20·430	36·701	57·131	122
C ..	3	8·808	16·588	25·396	15	8·622	17·191	25·813	17·430	33·779	51·209	111
P ..	4	8·111	15·762	23·873	16	8·301	15·931	24·232	16·412	31·365	48·105	104
P + G ..	5	12·512	22·222	34·734	17	13·648	21·073	34·721	26·160	43·295	69·455	151
P + C ..	6	8·558	15·894	24·452	18	10·349	17·346	27·695	18·907	33·240	52·147	113
S ..	7	7·039	14·138	21·177	19	8·392	15·573	23·965	15·431	29·711	45·142	98
S + G ..	8	14·700	25·658	40·358	20	13·717	21·294	35·011	28·417	46·952	75·369	163
S + C ..	9	10·608	18·662	29·270	21	9·629	18·082	27·711	20·237	36·744	56·981	123
P/2+S/2 ..	10	10·384	17·171	27·555	22	7·957	16·207	24·164	18·341	33·378	51·719	112
P/2+S/2+G..	11	13·871	25·667	39·538	23	15·042	23·774	38·816	28·913	49·441	78·354	170
P/2+S/2+C..	12	10·332	19·117	29·449	24	9·916	18·611	28·527	20·242	37·728	57·976	126

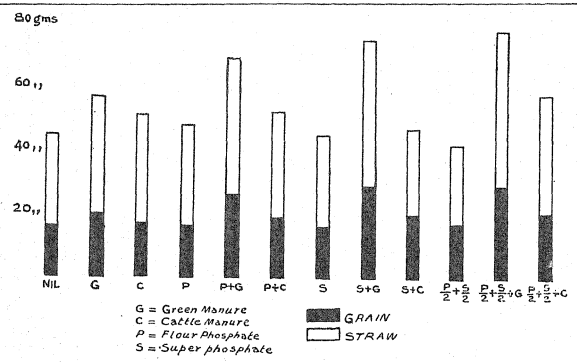


FIG. 7. Showing yield of paddy grain and straw in pot experiments to determine the availability of phosphate by green manure and cattle manure.

Discussion of results.

1. Taking the yield of total dry produce in no-manure pots as standard 100, flour phosphate gives 104 and superphosphate 98, showing that insoluble or soluble phosphate alone gives no increased crop.

2. While green manure pots give an yield of 122, flour phosphate + green manure give 151, superphosphate + green manure 163 and half flour phosphate + half superphosphate + green manure 170, showing that both insoluble and soluble phosphates respond in the soil when green manure is present.

3. While cattle manure pots give an yield of 111, flour phosphate + cattle manure give 113, superphosphate + cattle manure 123, and half flour phosphate + half superphosphate + cattle manure 126, showing that the phosphate respond somewhat in the soil with cattle manure, but not to the same extent as with green manure.

4. Fig. 7 brings out the inferences drawn here.

(*e*) AVAILABILITY OF FLOUR PHOSPHATE MEASURED BY THE GROWTH OF PADDY PLANTS IN POTS WITH AND WITHOUT GREEN MANURE AND WITH INCREASING QUANTITIES OF PHOSPHATE.

Pot Experiments—Series II (1916-17).

Experiments in other countries[1] generally indicate that, with increased application of ground mineral phosphate, increased crop is obtained up to a certain limit. An experiment was started to test the point with reference to the Trichinopoly phosphate.

Puddled soil equal to 12,000 grm. of air dry paddy soil were put in pots fourteen inches high and twelve inches broad, and flour phosphate and green manure were added as follows :—

Pot No.	Nature of experiment	Pot No.	Nature of experiment
1	P 250 lb. flour phosphate per acre ..	7	P +G 250 lb. flour phosphate and 5,000 lb. of green manure.
2	2P 500 „ „ „ „ „ ..	8	2P +G 500 lb. flour phosphate and 5,000 lb. of green manure.
3	3P 750 „ „ „ „ „ ..	9	3P +G 750 lb. flour phosphate and 5,000 lb. of green manure.
4	4P 1,000 „ „ „ „ „ ..	10	4P +G 1,000 lb. flour phosphate and 5,000 lb. of green manure.
5	5P 1,250 „ „ „ „ „ ..	11	5P +G 1,250 lb. flour phosphate and 5,000 lb. of green manure.
6	6P 1,500 „ „ „ „ „ ..	12	6P +G 1,500 lb. flour phosphate and 5,000 lb. of green manure.

[1] *Jour. Agri. Res.*, Vol. VI, No. 13, 1916, pp. 485-514.

The pots were attended to in the same manner as those of the first series, except that, as the pots were bigger, 7 seedlings were left in each pot. The results are tabulated in Table XIII and graphically represented in the charts in Fig. 8.

TABLE XIII.

No. of pot	Nature of experiment	Dry weight of			Average	Relative increase of yield in dry produce (green manure as standard)
		Grain	Straw	Total		
		grm.	grm.	grm.	grm.	
1	P	12·258	18·904	31·162	29·021	90
2	2 P	9·612	18·735	28·347		
3	3 P	10·770	18·854	29·624		
4	4 P	11·022	17·564	28·586		
5	5 P	9·912	17·444	27·356		
6	6 P	11·167	17·884	29·051		
7	P + G	17·822	31·038	48·860	49·691	154
8	2 P + G	21·135	33·314	54·449		
9	3 P + G	18·301	26·223	44·524		
10	4 P + G	20·774	34·541	55·315		
11	5 P + G	18·347	29·765	48·112		
12	6 P + G	19·278	27·610	46·888		
	Calculated from 1st series of experiments for 7 plants.					
1 + 13	Nil ..	11·099	21·182	32·281	32·281	100
2 + 14	G ..	14·301	25·691	39·992	39·992	121

No manure and *green manure* pots were not included in this series, in which each pot contained seven plants. For purposes of comparison with the figures in Table XIII, the yields for these pots were calculated from Table XII, Series I, in which each pot had only five plants.

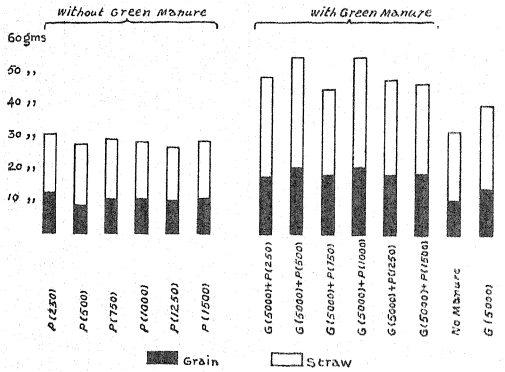


FIG. 8. Showing yield of paddy grain and straw in pot experiments with increasing quantities of flour phosphate with and without green manure.

Discussion of results.

1. Flour phosphate, by itself, is in no way advantageous unless supplemented with green manure.

2. Green manure renders the flour phosphate available by giving a relative yield of 154 against 121 given by green manure only and 90 given by flour phosphate only.

3. Increased quantities of flour phosphate, with and without green manure, give no increased cropping.

(*f*) AVAILABILITY OF FLOUR PHOSPHATE MEASURED BY THE GROWTH OF PADDY PLANTS IN POTS, NITROGEN ALSO BEING SUPPLIED.

Pot Experiments—Series III (1917–1922).

The experiments detailed in the previous section showed that, while green manure generally increased the availability of flour phosphate,

increased application of phosphatic manure gave no increased cropping contrary to the experience of other investigators. It was then surmised that, in addition to phosphoric acid, nitrogen might be a limiting factor in crop production. To test this point, the following series of experiments were started in 1917.

Paddy soil from M Block of the Central Farm, which contained 0·0384 per cent. of total phosphoric acid, 0·0066 per cent. of available phosphoric acid and 0·071 per cent. of nitrogen, was used for growing paddy, and the pots were filled as follows :—

(G=5,000 lb. green manure, P=250 lb. flour phosphate, N=224 lb. ammonium sulphate, per acre.)

Phosphate series	Nitrogen series	Green manure series	Green manure + nitrogen series
Nil.	N	G	G + N
P	N + P	G + P	G + N + P
2P	N + 2P	G + 2P	G + N + 2P
4P	N + 4P	G + 4P	G + N + 4P
8P	N + 8P	G + 8P	G + N + 8P

Experiments in different seasons.

1917–18. *Experiment I.*

The results of experiments carried out this year are shown in Table XIV and illustrated by Fig. 9.

TABLE XIV.

Showing availability of flour phosphate measured by growth of paddy in pots—7 plants per pot.

(P=250 lb. flour phosphate, G=2,000 lb. green manure, N=224 lb. ammonium sulphate, per acre.)

No. of pot	Nature of experiment	Dry weight of paddy			Relative yield of series	Relative yield of manured pots (green manure as standard)
		Grain	Straw	Total		
	Phosphate series	grm.	grm.	grm.		
1	Nil	0·690	9·610	10·300	100	20
2	P	1·918	9·670	11·588	113	
3	2P	2·770	11·294	14·064	137	
4	4P	3·533	11·091	14·624	142	
5	8P	3·625	12·692	16·317	158	
	Average of phosphate pots 2, 3, 4 & 5 ..	2·962	11·187	14·148	137	28
	Nitrogen series					
6	N	14·322	17·813	32·135	100	64
7	N & P	20·123	20·005	40·128	125	
8	N & 2P	21·638	24·210	45·848	143	
9	N & 4P	23·955	23·626	47·581	148	
10	N & 8P	24·756	24·764	49·520	154	
	Average of nitrogen and phosphate pots 7, 8, 9 & 10	22·618	23·151	45·769	142	92
	Green manure series					
1	G	20·687	29·404	50·091	100	100
2	G & P	32·743	34·000	66·743	133	
3	G & 2P	31·236	35·920	67·156	134	
4	G & 4P	38·326	45·173	83·499	167	
5	G & 8P	37·606	40·494	78·100	156	
	Average of green manure and phosphate pots 2, 3, 4 & 5 ..	34·976	38·898	73·874	147	147

TABLE XIV—*concld.*

No. of pot	Nature of experiment	Dry weight of paddy			Relative yield of series	Relative yield of manured pots (green manure as standard)
		Grain	Straw	Total		
	Green manure and nitrogen series	grm.	grm.	grm.		
6	G & N	32·650	40·033	72·683	100	145
7	G & N & P ..	34·497	41·618	76·115	105	
8	G & N & 2P ..	40·787	51·912	92·699	128	
9	G & N & 4P ..	38·681	45·632	84·313	116	
10	G & N & 8P ..	41·402	45·774	87·176	120	
	Average of green manure and nitrogen and phosphate pots 7, 8, 9 & 10	38·842	46·234	85·076	117	165

Remarks. The plants in the series of pots without nitrogen or green manure showed poor growth from the beginning and were sickly in appearance; the soil was very hard under the puddle and roots did not develop properly. Green manure gives better results than nitrogen, but green manure+nitrogen pots are the best of all. There is a certain amount of increase in crop with increasing quantities of phosphate in all the series.

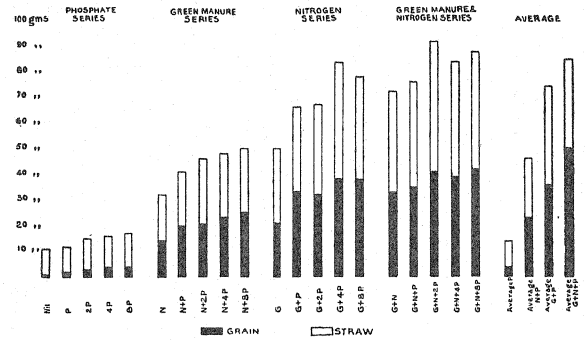


FIG. 9. Showing yield of paddy grain and straw in pot experiments to determine the availability of flour phosphate with and without nitrogen and with and without green manure.

1918–19, 1919–20.

The experiments in these two years were vitiated on account of the damage done to the earheads at the ripening stage by squirrels which came in through the ceiling or small openings in the wire-netting roof of the pot culture house.

1920–21. *Experiment II.*

The pots were now protected from squirrels by being placed in special wire-netting cages. The results are shown in Table XV and illustrated by Fig. 10 and by the photographs of paddy plants in Plate III.

TABLE XV.

Showing dry weight of paddy grain and straw in pot experiments, nitrogen being also supplied—7 plants per pot.

(P=250 lb. flour phosphate, G=2,000 lb. green manure, N=224 lb. ammonium sulphate, per acre).

No. of pot	Nature of experiment	No. of tillerings	Dry weight of paddy			Relative yield in each series	Relative yield of manured pots (green manure as standard)
			Grain	Straw	Total		
	Phosphate series		grm.	grm.	grm.		
6	Nil	19	7·425	13·310	20·735	100	39
7	P	30	10·651	15·545	26·196	126	
8	2P	30	11·867	15·240	27·107	131	
9	4P	32	12·776	14·020	26·796	129	
10	8P	33	12·318	12·150	24·468	118	
	Average of phosphate pots 7, 8, 9 & 10 ..	31	11·902	14·239	26·141	126	50
	Nitrogen series						
1	N	28	13·054	20·862	33·916	100	62
2	N & P	29	13·200	19·240	32·440	96	
3	N & 2P	30	16·652	21·265	37·917	112	
4	N & 4P	31	18·136	21·305	39·441	117	
5	N & 8P	29	20·740	22·505	43·045	127	
	Average of nitrogen and phosphate pots 2, 3, 4 & 5	30	17·132	21·079	38·211	113	73
	Green manure series						
16	G	39	24·603	28·100	52·703	100	100
17	G & P	42	24·716	33·510	58·226	111	
18	G & 2P	47	26·512	33·330	59·842	113	
19	G & 4P	50	28·119	35·195	63·314	120	
20	G & 8P	50	32·385	35·115	67·500	128	
	Average of green manure and phosphate pots 17, 18, 19 & 20 ..	47	27·933	34·288	62·221	118	118

TABLE XV—*concld.*

No. of pot	Nature of experiment	No. of tillerings	Dry weight of paddy			Relative yield in each series	Relative yield of manured pots (green manure as standard)
			Grain	Straw	Total		
	Green manure and nitrogen series		grm.	grm.	grm.		
11	G & N	52	26·809	42·895	69·704	100	132
12	G & N & P ..	60	27·072	47·470	74·542	107	
13	G & N & 2P ..	62	32·331	47·765	80·096	115	
14	G & N & 4P ..	62	36·810	53·715	90·525	130	
15	G & N & 8P ..	68	36·738	56·540	93·278	133	
	Average of green manure and nitrogen and phosphate pots 12, 13, 14 & 15	63	33·238	51·372	84·610	121	161

Remarks. Judged by the healthiness in appearance and robustness of crop during growth, by the number of tillers and by the yield of grain and straw, flour phosphate by itself gives the lowest yield, nitrogen series is better, green manure series is better still, but green manure + nitrogen series is the best of all. Increasing quantities of phosphate give increased crop in all series except the flour phosphate series, but when nitrogen is also applied, this increase is more evident. Nitrogen is a limiting factor in the soil, and flour phosphate shows its best effects when nitrogen is supplied along with green manure.

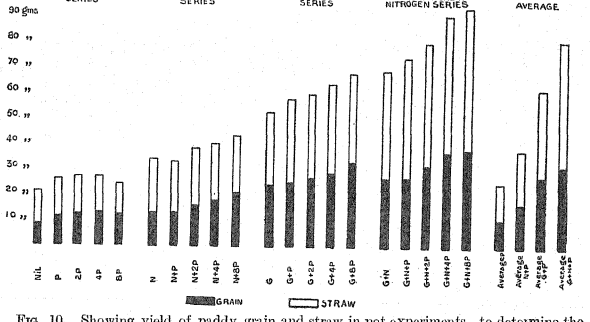


FIG. 10. Showing yield of paddy grain and straw in pot experiments to determine the availability of flour phosphate with and without nitrogen and with and without green manure.

1921–22. *Experiment* III.

The same experiment was repeated this year with greater precautions in duplicate; but, as 40 pots of the same size were not available, the duplicates were mostly grown in small pots, 8 seedlings being left in each of the originals and 6 seedlings in each of the duplicates. The results are tabulated in Table XVI and illustrated by Fig. 11.

TABLE XVI.

Showing availability of flour phosphate, measured by the growth of paddy in pots—14 plants.
(P=250 lb. flour phosphate, G=2,000 lb. green manure, N=224 lb. ammonium sulphate, per acre.)

No. of pot	Nature of experiment	ORIGINAL—8 PLANTS PER POT					DUPLICATE—6 PLANTS PER POT					ORIGINAL AND DUPLICATE —14 PLANTS			Relative yield in each series	Relative yield of manured pots (green manure as standard)
		No. of pot	No. of tillerings	WEIGHT OF DRY PRODUCE			No. of pot	No. of tillerings	WEIGHT OF DRY PRODUCE							
				Grain	Straw	Total			Grain	Straw	Total	Grain	Straw	Total		
	Phosphate series			grm.	grm.	grm.			grm.	grm.	grm.	grm.	grm.	grm.		
1	Nil	1	8	6·49	9·15	15·64	21	8	3·55	8·21	11·76	10·04	17·36	27·40	100	38
2	P	2	10	9·51	10·29	19·80	22	8	7·24	9·03	16·27	16·75	19·32	36·07	132	
3	2P	3	10	6·36	12·60	18·96	23	11	8·13	11·13	19·26	14·49	23·73	38·22	139	
4	4P	4	14	8·26	15·55	23·81	24	14	8·14	14·17	22·31	16·40	29·72	46·12	168	
5	8P	5	17	14·40	18·13	32·53	25	15	6·77	14·81	21·58	21·17	32·94	54·11	200	
	Average of phosphate pots 2, 3, 4 & 5	..	..	..	..	..	..	..	..	..	..	17·20	26·43	43·63	..	61
	Nitrogen series															
6	N	6	17	15·19	20·01	35·20	26	14	10·93	13·92	24·85	26·12	33·93	60·05	100	84
7	N & P	7	19	16·69	21·51	38·20	27	15	12·57	15·11	27·68	29·26	36·62	65·88	109	
8	N & 2P	8	20	16·38	21·72	38·10	28	15	17·05	17·90	34·95	33·43	39·62	73·05	120	
9	N & 4P	9	18	17·70	21·66	39·36	29	18	15·10	19·16	34·26	32·80	40·82	73·62	121	
10	N & 8P	10	16	18·70	22·38	41·08	30	23	17·32	20·67	37·99	36·02	43·05	79·07	132	
	Average of nitrogen phosphate pots 7, 8, 9 & 10	..	..	..	..	..	..	..	..	..	..	32·88	40·03	72·91	..	102
	Green manure series															
11	G	11	19	19·36	24·51	43·87	31	15	9·80	18·09	27·89	29·16	42·60	71·76	100	100
12	G & P	12	23	23·49	29·52	53·01	32	17	13·82	20·90	34·72	37·31	50·42	87·73	122	
13	G & 2P	13	22	22·40	27·47	49·87	33	17	12·70	20·02	32·72	35·10	47·49	82·59	115	
14	G & 4P	14	21	22·65	27·72	49·77	34	16	11·18	21·77	32·95	33·23	49·49	82·72	115	
15	G & 8P	15	24	25·64	32·63	58·27	35	17	14·32	23·57	37·89	39·96	56·20	96·16	134	
	Average of green manure and phosphate pots 12, 13, 14 & 15 ..	..	..	..	..	..	..	..	..	..	..	36·40	50·99	87·39	..	122

	Green manure and nitrogen series														
16	G & N	16	23	21·68	32·26	53·94	36	17	12·94	22·28	35·22	34·62	54·54	89·16	100	124
17	G & N & P ..	17	25	33·22	38·73	71·95	37	18	16·47	25·37	41·84	49·69	64·10	113·79	128	
18	G & N & 2P ..	18	27	32·93	38·19	71·12	38	19	19·32	26·83	46·15	52·25	65·02	117·27	132	
19	G & N & 4P ..	19	26	37·22	38·90	76·12	39	19	21·39	28·01	49·40	58·61	66·91	125·52	141	
20	G & N & 8P ..	20	26	35·26	44·80	80·06	40	19	22·07	29·51	51·58	57·33	74·31	131·64	148	
	Average of green manure and nitrogen and phosphate pots 17, 18, 19 & 20 ..	..	..	..	..	..	..	..	..	..	..	54·47	67·59	122·06	..	170

Remarks. This experiment corroborates the results obtained in the previous two experiments in that green manure + nitrogen series renders the flour phosphate more available than the other series. There is an increase in crop due to increased application of phosphate, but it is not uniform increase. At any rate, there is no increase commensurate with the increased cost of manure.

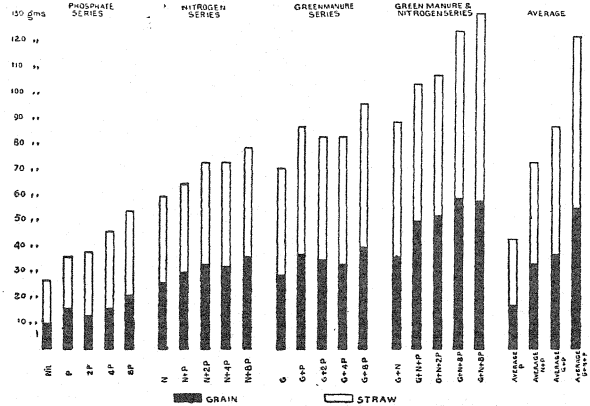


FIG. 11. Showing yield of paddy grain and straw in pot experiments to determine the availability of flour phosphate with and without nitrogen and with and without green manure.

TABLE XVII.

Abstract of Experiments I, II and III, showing availability of flour phosphate, measured by the growth of paddy in pots.

(P=250 lb. flour phosphate, G=2,000 lb. green manure, N=224 lb. ammonium sulphate, per acre.)

Serial No.	Nature of experiment	DRY WEIGHT OF PADDY IN GRM.												Relative yield of each series	Relative yield of each pot (green manure as standard)
		EXPERIMENT I			EXPERIMENT II			EXPERIMENT III			TOTAL				
		Grain	Straw	Total	Grain	Straw	Total	Grain	Straw	Total	Grain	Straw	Total		
	Phosphate series														
1	No manure	0·62	9·61	10·30	7·43	13·31	20·74	15·04	17·36	27·40	18·16	40·28	58·44	100	34
2	P	1·02	9·87	11·59	10·65	15·54	26·19	16·75	19·32	36 07	29·32	44 53	73·85	126	42
3	2P	2·77	11·29	14·06	11·87	15·24	27·11	14·49	23·73	38·22	29·13	50·26	79·39	136	46
4	4P	3·53	11·09	14 62	12·77	14·02	26·79	16·40	29·72	46·12	32·70	54·83	87·53	149	50
5	8P	3·63	12·69	16·32	12·32	12·15	24·47	14·17	32·94	54·11	37·12	57·78	94·90	163	54
	Phosphate series 2, 3, 4 & 5 ..	Total	11·85	44·74	56·59	47·61	56·95	104·56	68·81	105·71	174·52	128·27	207·40	335·67	..	..
		Average	2·96	11·19	14·15	11·90	14·24	26·14	17·20	26·43	43·63	32·07	51 85	83 92	144	48
	Nitrogen series														
6	N	14·32	17·81	32·13	13·05	20·86	33·91	26·12	33·03	60·05	53·49	72·60	126·09	100	72
7	N & P	20·12	20·01	40·13	13·20	19·24	32·44	29·26	36·62	65 88	62·58	75·87	138·45	110	79
8	N & 2P	21·64	24·21	45·85	16·65	21·27	37·92	33·43	39·62	73·05	71·72	85·10	156·82	124	90
9	N & 4P	23·95	23·63	47·58	18·14	21·30	39·44	32·80	40·82	73·62	74·89	85·75	160·74	128	92
10	N & 8P	24·76	24·76	49·52	20·54	22·51	43·05	36·02	43·05	79·07	81·32	90·32	171·64	136	98
	Phosphate + nitrogen series 7, 8, 9 & 10 ..	Total	90·47	92·61	183·08	68·53	84·32	152·85	131·51	160·11	291·62	290·51	337·04	627·55	..	..
		Average	22·62	23·15	45·77	17·13	21·08	38·21	32·88	40·03	72·91	72·63	84·26	156·89	124	90
	Green manure series														
11	G	20·69	29·40	50·09	24·60	28·10	52·70	29·16	42·60	71·76	74·45	100·10	174·55	100	100
12	G & P	32·74	34·00	66·74	24·72	33·51	58·23	37·31	59·42	87·73	94·77	117·93	212·70	122	122
13	G & 2P	31·24	35·92	67·16	26·51	33·33	59·84	35·10	47·49	82·59	92·85	116·74	209·59	120	120
14	G & 4P	38·33	45·17	83·50	28·12	35·19	63·31	33·23	49·49	82·72	99·68	129·85	229·53	131	131
15	G & 8P	37·61	40·49	78·10	32·39	35·11	67·50	38·06	56·20	96·16	108·06	131·80	241·76	138	138
	Green manure + phosphate series 12, 13, 14 & 15 ..	Total	139·92	155·58	295·50	111·74	137·14	248·88	145·60	203·60	349·20	397·26	496·32	893·58	..	..
		Average	34·98	38·90	73·88	27·94	34·28	62·22	36·40	50·90	87·30	99·32	124·08	223·40	128	128

	Green manure and nitrogen series														
16	G & N	32·65	40·03	72·68	26·80	42·00	69·70	34·62	54·54	89·16	94·07	137·47	231·54	100	132
17	G & N & P	34·50	41·62	76·12	27·07	47·47	74·54	49·69	64·10	113·79	111·26	153·19	264·45	114	151
18	G & N & 2P	40·79	51·91	92·70	32·33	47·77	80·10	52·25	65·02	117·27	125·37	164·70	290·07	125	166
19	G & N & 4P	38·68	45·63	84·31	36·81	53·72	90·53	58·61	66·91	125·52	134·10	166·26	300·36	130	172
20	G & N & 8P	41·40	45·77	87·17	36·74	56·54	93·28	57·33	74·31	131·64	135·47	176·62	312·09	135	179
	Green manure + nitrogen + phosphate series 17, 18, 19 & 20 ..	Total	155·37	184·93	340·30	132·95	205·50	338·45	217·88	270·34	488·22	506·20	660·77	1166·97	..	..
		Average	38·84	46·23	85·07	33·24	51·37	84·61	54·47	67·59	122·06	126·55	165·19	291·74	126	167

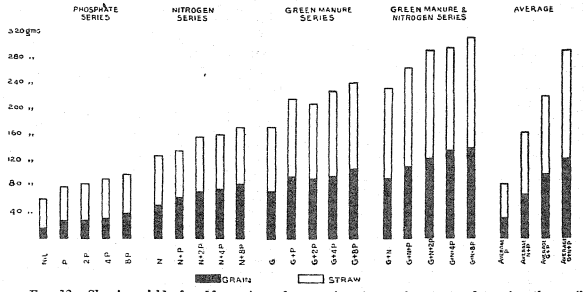


FIG. 12. Showing yield of paddy grain and straw in pot experiments to determine the availability of flour phosphate with and without nitrogen and with and without green manure.

General remarks and discussion of results.

Taking all the three experiments together, which were carried on in three different seasons, the following inferences may be drawn :—

1. There is an increased crop obtained with increasing applications of phosphate in each case. The average availability of phosphate in each series is not far from the availability when 500 lb. of flour phosphate have been applied, indicating that there is no object in applying larger quantities than 500 lb. per acre.

2. Next to no-manure pots, phosphate series, serial numbers 2 to 5, give the lowest yield.

3. The nitrogen series, numbers 6 to 10, give a better crop than the phosphate series, showing that nitrogen is a limiting factor.

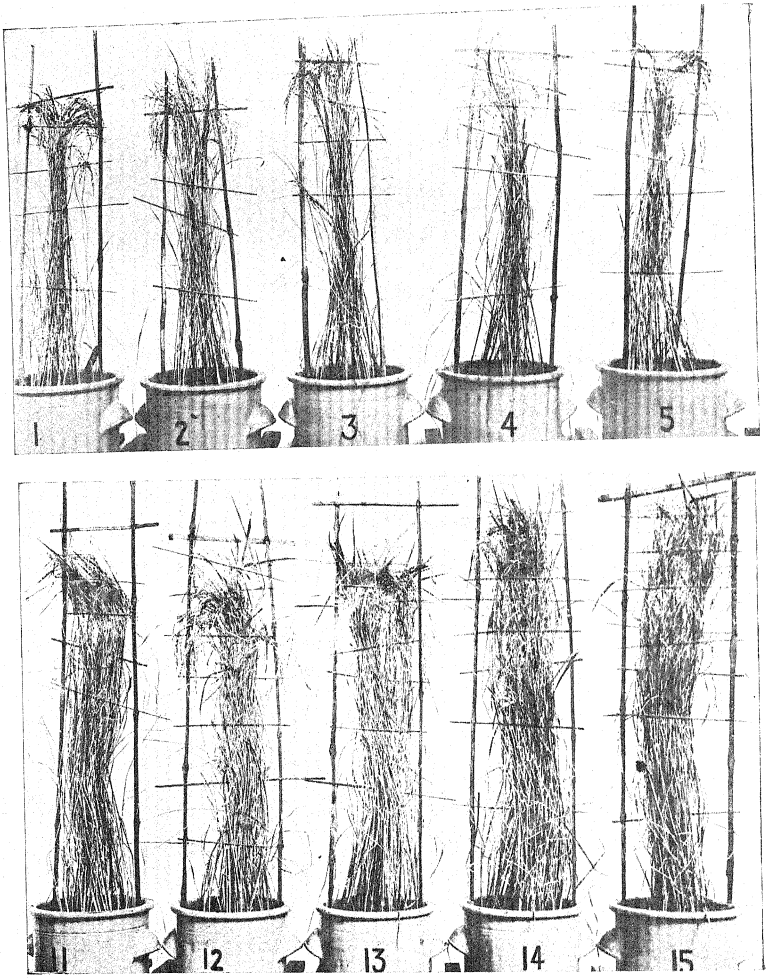
4. The green manure series, numbers 11 to 15, are better than nitrogen series.

5. Green manure + nitrogen series, numbers 16 to 20, uniformly give the best yields of all, showing that green manure alone or nitrogen alone cannot produce the maximum availability of flour phosphate. The available nitrogen of the ammonium sulphate apparently gives a start for the vegetative growth of the plants and the healthy adult plants are then able to utilize the phosphate rendered available by green manure.

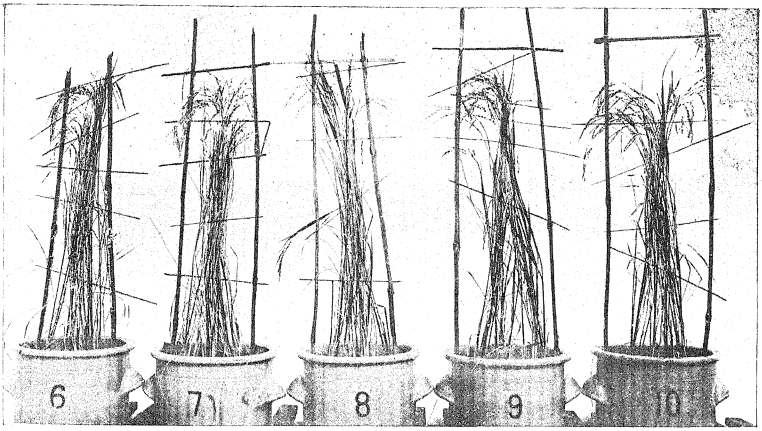
6. Taking green manure pots as standard 100, we find that the average yields of all the series are as follows :—

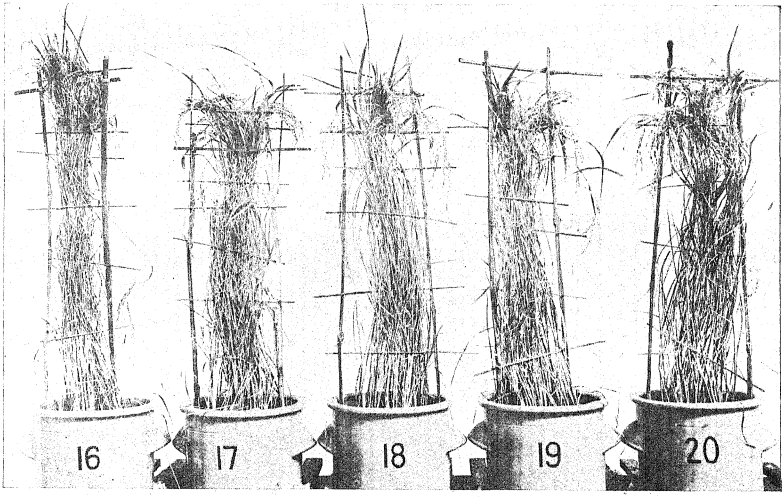
No manure	34
Phosphate only	48
Nitrogen only	72
Green manure only	100
Nitrogen + phosphate	90
Green manure + phosphate	128
Green manure + nitrogen	132
Green manure + nitrogen + phosphate	167

These experiments distinctly show that flour phosphate is rendered available to paddy under paddy soil conditions when green manure is ploughed in, and the results are more striking when nitrogen is supplied as well.



Paddy plants in pots manured with sulphate of ammonia, Trichinopoly





flour phosphate and *dhaincha* leaves separately and in combinations.

(*g*) Availability of flour phosphate measured by the growth of paddy on field scale in conjunction with green manure.

However carefully designed and carried out, it was realized that there were several limitations to the formation of ideal paddy soil conditions in the experiments in pots; and it was considered necessary to determine the efficacy of green manure in making flour phosphate available to paddy under swampy conditions on field scale in ryots' lands, and this was done with the co-operation of district agricultural officers. Apart from these field experiments, the results obtained at the Manganallur Agricultural Station, bearing on the subject, are also appended.

I. Co-operative field experiments.

Co-operative experiments on field scale for the trial of flour phosphate on ryots' lands were begun in 1918–19 and, after an interval of three years, taken up again in 1921–22. Detailed instructions were issued to the agricultural demonstrators for the selection of sites, the laying out of plots, the application of manure, the transplanting of seedlings and the harvesting of crop, the object kept in view being the comparison of the growth of paddy plants of a long duration variety in an acre of land divided into 10 ten-cent strips uniformly puddled with green manure, the alternate even strips receiving flour phosphate in addition at the rate of 250 lb. per acre. These detailed instructions were not and, in some localities, could not, however, be followed; and it is desirable to point out how very difficult it is to carry on field experiments on ryots' lands. For instance, where suitable land was available, the persons, who first promised to place their land for experimental purposes, either withdrew their offer later or had begun to plant without notice. Some lands were freely offered for experiment, which happened to be particularly alkaline. In some cases, cattle manure had also been applied, or cattle or sheep penned in the fields, so that the experiment cannot be said to be one calculated to test the effect of green manure on flour phosphate. Again, deviation had to be made as regards the area of each plot, as the land available could not conveniently be divided into 10 ten-cent plots, even in Government agricultural stations. Where several small contiguous fields were alone available, half of each field received flour phosphate and green manure, while the other half received green manure only. In most cases, it was possible to get the outskirts

near the bunds put outside the experimental area, but this was not done in all cases. The season in 1918-19 was one of the most unfavourable for paddy cultivation in Southern India, and, in not a few cases, the manure was applied late to a short duration variety of paddy, leaving little time for the dissolving of the phosphate. Lastly, while the straw yields obtained in Government farms were generally air dry weights, the straws on private lands were either not fully dried or they were not cut uniformly, the prevailing practice in the Tanjore District, for instance, being to leave a foot and more of the straw as a stubble in the field. The figures about yield of straw, not having been obtained in a uniform manner, have been omitted in the statements given below.

In spite of all the above mentioned drawbacks and difficulties, it has to be said to their credit that most of the agricultural subordinate officers entered into the spirit of the experiment and submitted very careful notes, and the author is thankful to them, as also to the gentlemen who placed their lands at his disposal and to the Deputy Directors of Agriculture for their sympathetic co-operation.

Table XVIII gives general information regarding the field experiments of the two seasons, Table XIX gives the yields of grain and indicates the limitations of the experiments, and Table XX is compiled to show the yields of grain in plots which contain less than 0·01 per cent. of available phosphoric acid and are, therefore, distinctly poor in that ingredient.

TABLE XVIII.

Showing details of co-operative field experiments with flour phosphate.

No. of experiment	Season	Locality	Sources of irrigation	Circle	GREEN MANURE		GREEN MANURE AND FLOUR PHOSPHATE	
					No. of plots	Total area of plots in cents	No. of plots	Total area of plots in cents
1	1918–19	Kilmanur, N. Arcot Dt.	Tank fed by R. Palar ..	IV Circle	4	32	4	32
2	1918–19	Nazarathpettai, Chingleput Dt.	Tank ..		5	50	5	50
3	1918–19	Kayanallur, Chingleput Dt.	Do. ..		5	50	5	50
4	1918–19	Illedu, Chingleput Dt.	Do. ..		5	50	5	50
5	1918–19	Ettapur, Salem Dt	R. Vasishta and wells	VIII Circle	5	50	5	50
6	1921–22	Do. ..	Do. ..		5	33	5	33
7	1921–22	Danishpet, Salem Dt.	Tank ..		4	40	4	40
8	1921–22	Kodiveri, Coimbatore Dt.	R. Bhavani		..	53	..	50
9	1921–22	Dalavoypatnam, Coimbatore Dt.	R. Amaravati.		8	47	8	47
10	1921–22	Elandangudy, Tanjore Dt.	R. Cauvery	V Circle	7	28	7	28
11	1921–22	Avarani, Tanjore Dt.	Do. ..		4	40	4	40
12	1921–22	Tirupalathurai, Trichinopoly Dt.	Do. ..		4	40	4	40
13	1919–20	Central Farm, Coimbatore	Tank fed by R. Noyyal	Central Farm, Coimbatore	4	34·5	4	35·3
14	1920–21	Do. ..	Do. ..		4	34·5	4	35·3
15	1921–22	Do. ..	Do. ..		4	34·5	4	35·3
16	1922–23	Do. ..	Do. ..		4	34·5	4	35·3

TABLE XIX.

Showing yield of paddy grain in co-operative field experiments with flour phosphate.

No. of experiment	Locality	Soil analysis			Yield of grain per acre		Difference in yield of grain due to phosphate (+ or −)		Remarks
		Total phosphoric acid	Available phosphoric acid	Nitrogen	Green manure	Green manure + flour phosphate	Quantity	Percentage	
		per cent.	per cent.	per cent.	lb.	lb.	lb.	per cent.	
1	Kilmanur ..	Not analysed.			1,573	1,577	+4		(a)
2	Nazarethpettai ..	Do.			2,750	2,690	−60	−2	
3	Kayanallur ..	Do.			2,180	2,202	+22	+1	
4	Illedu	Do.			3,360	3,376	+16		
5	Ettapur ..	0·041	0·006	0·070	2,138	2,550	+412	+19	
6	Do.	0·041	0·008	0·125	3,759	4,060	+301	+8	
7	Danishpet ..	0·035	0·0017	0·096	3,603	3,879	+276	+8	
8	Kodiveri ..	0·081	0·022	0·062	2,359	2,352	−7		(b)
9	Dalavoypatnam ..	0·178	0·035	0·074	2,362	2,508	+144	+6	(c)
10	Elandangudy ..	0·030	0·002	0·053	2,102	2,441	+338	+16	
11	Avarani ..	0·030	0·003	0·058	2,097	2,303	+206	+10	
12	Tiruppalathurai ..	0·162	0·085	0·072	2,860	2,800	−60	−2	(d)
13	Central Farm, Coimbatore	0·038	0·006	0·071	2,031	2,050	+19	+1	
14	Central Farm, Coimbatore	0·038	0·006	0·071	2,013	2,208	+195	+10	(e)
15	Central Farm, Coimbatore	0·038	0·006	0·071	2,557	2,778	+221	+9	
16	Central Farm, Coimbatore	0·038	0·006	0·071	1,907	2,089	+181	+9	

(*a*) Season was most adverse for paddy. Short duration variety was planted two months later than usual season. Cattle manure was also applied along with green manure.

(*b*) In a comparative experiment laid down side by side, bonemeal + groundnut cake did not give any increased yield.

(*c*) Field was uneven, and the plot yields were extremely variable.

(*d*) Fourteen cartloads of cattle manure were applied and 300 cattle also penned.

(*e*) Standardized plots.

Discussion of results.

Of the 16 field experiments shown in Table XVIII, half the number (Expt. Nos. 1, 2, 3, 4, 8, 9, 12 and 13) give little or no increase of grain as a result of the application of flour phosphate. Of these, Nos. 1 to 4 may be discarded for the reasons specified in the footnote to Table XIX. Nos. 8 and 12 were well supplied with total and available phosphoric

acid. The soil of No. 9 is rich in available phosphoric acid and still gives an increased yield of 6 per cent. in favour of phosphate plots, but the yields of individual plots are most erratic, showing that the plots are far from uniform. As regards No. 13, it is possible that the phosphate did not get dissolved in the soil in sufficient quickness to be available in the first year, but gave a consistent increase in three subsequent years to the extent of 9 to 10 per cent.

As regards the other half of the Experiments (Nos. 5, 6, 7, 10, 11, 14, 15 and 16), there is a substantial increase in the yield of grain, ranging from 182 to 412 lb. per acre and with an average of 267 lb. in favour of flour phosphate, as shown in Table XX ; and it will be noticed that the soils in all these experiments, though fairly well supplied with nitrogen, contained less than 0·01 per cent. of available phosphoric acid. Expressed in percentage, this increased yield varies from 8 to 19 per cent. and amounts to 10·5 per cent. on the average, due to the application of flour phosphate and green manure, over the yield of plots manured with green manure only. It is significant that this figure is nearly the same as was obtained in the standardized plots of the Central Farm in three consecutive years.

TABLE XX.

Showing increased yield of paddy grain in co-operative field experiments in soils containing less than 0·01 per cent. available phosphoric acid.

Experiment No.	Locality	Yield of grain per acre		Increased yield due to flour phosphate		Remarks
		Green manure	Green manure + flour phosphate	Quantity	Percentage	
		lb.	lb.	lb.		
5	Ettapur ..	2,138	2,550	412	19	
6	Ettapur ..	3,759	4,060	301	8	
7	Danishpet ..	3,603	3,879	276	8	
10	Elandangudy ..	2,103	2,441	338	16	
11	Avarani ..	2,097	2,303	206	10	
14	Central Farm ..	2,013	2,208	195	10	
15	Central Farm ..	2,557	2,778	221	9	
16	Central Farm ..	1,907	2,089	182	9	
	AVERAGE ..	2,522	2,789	267	10·5	

II. *Experiments at the Manganallur Agricultural Station.*

This station was maintained on lease for ten years from 1912 with the object of studying the cultivation of paddy in the Cauvery delta and of finding out suitable manures for the same. The soil consisted largely of silt and clay, to the extent of over 70 per cent., and was fairly representative of the extensive low-lying lands of Tanjore, being comparatively poor in plant-food, containing on an average only 0·044 per cent. of nitrogen, 0·04 per cent. of total phosphoric acid and 0·0026 per cent. of available phosphoric acid. Careful experiments were carried on at this agricultural station by the Deputy Director of Agriculture, V Circle, some on his own initiative, some under instructions from the Government Agricultural Chemist and a few in consultation with me, to test the value of different manures for paddy land in Tanjore, from 1914 onwards until the station was closed two years ago, flour phosphate being one of the phosphatic manures chosen for trial. The results have been published, with copious charts, as *Bulletin No. 85 of the Madras Agricultural Department*, and the following extracts are taken from it.

TABLE XXI.

Statement showing relative yield of paddy grain and straw on the application of different phosphatic manures. (Average of four years.)

Manure	Application of 20 lb. of phosphoric acid		Application of 60 lb. of phosphoric acid	
	Grain	Straw	Grain	Straw
Check	100	100	100	100
Bone super	116	109	128	129
Bonemeal	113	108	117	117
Flour phosphate ..	113	103	110	111

Remarks. It was found that mineral phosphate showed gradual increase in yield from year to year and, though not immediately available, it became available fairly quickly. The average increase in yield, with an application of flour phosphate containing 20 lb. of phosphoric acid, was 13 per cent. over the check plots, the same as was obtained by the application of bonemeal containing the same quantity of phosphoric acid, and no advantage was gained by the application of larger quantities. This last statement is confirmed by another experiment in which 300 lb. and 500 lb. of bonemeal and flour phosphate were applied to different plots.

TABLE XXII.

Statement showing effect of different quantities of phosphatic manures.
(Average of four years.)

Manure	AVERAGE RELATIVE YIELD FOR FOUR YEARS	
	Grain	Straw
Check	100	100
Bonemeal 500 lb. ..	134	145
Bonemeal 300 ,, ..	122	127
Flour phosphate 500 ,, ..	115	124
Flour phosphate 300 ,, ..	114	123

Remarks. Bonemeal was distinctly superior to flour phosphate, which has to be ascribed to the organic matter and nitrogen it contained. The flour phosphate gave, however, an increase of 14 to 15 per cent. of grain and 23 to 24 per cent. of straw over the check plot.

In another experiment, flour phosphate containing 20 lb. of phosphoric acid was applied for four years, and the residual effects were noted in succeeding years.

TABLE XXIII.

Statement showing residual effect of manures supplying 20 lb. of phosphoric acid.

Manure	AVERAGE YIELD 1914–15 TO 1917–18		RESIDUAL EFFECT—RELATIVE YIELD					
			1918–19		1919–20		Average	
	Grain	Straw	Grain	Straw	Grain	Straw	Grain	Straw
Check	100	100	100	100	100	100	100	100
Bone super ..	116	109	101	104	112	124	106	110
Bonemeal ..	113	108	114	132	113	120	114	129
Flour phosphate ..	113	103	107	112	120	131	113	117

The results obtained at the Manganallur Agricultural Station show, beyond doubt, that flour phosphate is rendered available by the green manure, corroborating the results obtained by the author, and that there is also a residual effect to the extent of 13 per cent. increase in grain in the third year after application of manure.

Manure mixture for Tanjore paddy soils.

Before closing, a word may be said about special *manure mixtures* for Tanjore soils, the investigation of which the author has undertaken as a sequence of his work on flour phosphate. During the progress of these investigations, a few experiments were done in 1918 on the rate at which different organic manures, like castor cake, groundnut cake and fish guano, rendered the flour phosphate available; and it was then found that from 20 to 50 per cent. of the phosphoric acid contained in the flour phosphate was soluble in 1 per cent. citric acid at 65°C. in soil composts kept in the sun in a puddled condition for ten days. Based on these experiments, a manure mixture was adopted by the Deputy Director which has found general favour in the district.

Field and pot experiments have been started from 1923 with the object of evolving suitable manure mixtures based on current prices. Useful results have been obtained so far, but the experiments are being repeated and are in progress this year.

PART III.

SUMMARY AND CONCLUSIONS.

A deposit of phosphatic nodules, containing 56 to 59 per cent. of tricalcium phosphate, 17 to 20 per cent. of calcium carbonate, 7 per cent. of iron oxide and alumina and 7 per cent. of silica, is present in the cretaceous formation in the Trichinopoly District, lying on the surface and imbedded in soft yellow clay. A full description of the locality is given.

2. The quantity is estimated at about 8 million tons, to a depth of 200 feet, for a length of over ten miles and a breadth of over one mile.

3. Paddy soils in the adjoining Tanjore District, of which over a million acres are under the crop, are largely deficient in total and available phosphoric acid, as revealed by the soil survey of the district, and a suitable phosphatic manure is necessary for these soils. It is desirable to get this supply from the locality of phosphatic nodules, which is so near and accessible.

4. The nodules are not suitable for superphosphate manufacture as the high percentage of calcium carbonate and iron oxide and alumina will result in waste of acid.

5. The conversion of tricalcic phosphate of the nodules into dicalcic phosphate is not feasible at present, but may be worthy of consideration, if and when the hydro-electric scheme to utilize the water power in the Kollemalai and Pachamalai Hills at all takes shape.

6. The nodules have, therefore, to be applied to the soil in a finely powdered condition, and it is shown by various experiments that the flour

phosphate slowly gets dissolved in the soil water and becomes available to the paddy plant.

7. Water containing carbonic acid dissolves tricalcium phosphate to an appreciable extent ; and the greater the quantity of carbonic acid acting on it, the more does it dissolve.

8. The decomposition of organic matter, in the form of green manure, under swampy conditions of cultivation, results in the formation of sufficient quantities of carbonic acid to convert appreciable quantities of tricalcium phosphate into dicalcium and monocalcium phosphates which are soluble in soil water.

9. The availability of phosphate in composts made with green manure and cattle manure was measured by its solubility in different conventional reagents ; and the solubility was found to be greatest in one week's composts and to decrease in longer kept composts. At the same time there was considerable diminution in weight in the organic matter of such composts, showing that the organic matter had largely decomposed during the composting period. This reaction is explained on the supposition that soluble phosphate continues to be formed in long kept composts, but that it is speedily reverted by the lime present in the compost. For this reason, it is suggested that flour phosphate may be applied along with green manure at the time of puddling, so that the reversion may take place round soil particles in the soil, in preference to its taking place in the manure heap.

10. Measured by the yield of paddy grown in pots, it is found that organic matter renders the phosphate available and produces increased crop ; and that green manure acts much better in this respect than cattle manure.

11. Increased quantities of phosphate did not give increased cropping, both with and without green manure, unless nitrogen, which was a limiting factor in the soil, was also supplied.

12. In a series of experiments in pots, conducted over a number of years, the average relative yield of dry produce was as follows, taking green manure as standard :—

No manure	34
Phosphate only	48
Nitrogen only	72
Green manure only	100
Nitrogen + phosphate	90
Green manure + phosphate	128
Green manure + nitrogen	132
Green manure + nitrogen + phosphate	167

The results show that, when sufficient nitrogen is present in the soi green manure renders the flour phosphate available to the paddy plant.

13. In the above experiments, increased applications of flour phosphate gave increased cropping up to a certain limit, but not commensurate with the cost of the increased quantity of manure applied. Generally 250 to 500 lb. per acre every year may be considered sufficient.

14. Co-operative field experiments on ryots' lands done in 1918–19 were discarded for reasons mentioned in the memoir, chiefly because the season was most unfavourable for paddy that year. As regards those done in 1921–22, soils containing over 0·01 per cent. of available phosphoric acid do not respond to flour phosphate, but in soils containing less than 0·01 per cent. of available phosphoric acid, flour phosphate with green manure gives increased crops varying from 8 to 19 per cent. over green manure only, with an average of 10·5 per cent.

15. Field experiments at the Manganallur Agricultural Station and on the standardised plots in the Central Farm, Coimbatore, for four years, give an increased yield of grain, in favour of flour phosphate + green manure, of 13 per cent. and 9 per cent., respectively, over the yield in check plots.

16. The residual value of flour phosphate, as measured at the Manganallur Agricultural Station, is appreciable.

Recommendations.

1. The supply of phosphatic manure for the Tanjore delta should be more largely obtained from the phosphatic nodules which should be crushed as fine as possible, the manure being priced as much for its degree of fineness, as for its content of phosphoric acid.

2. The flour phosphate may be either ploughed in with green manure, when the land is prepared for cultivation, or may be mixed in suitable proportions with organic manures, like oil cakes, to produce special manure mixtures, which should be sold and purchased under a guarantee.

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